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JUL 79 C S KORTOVICH, J M MARDER F33615-76-C-5105

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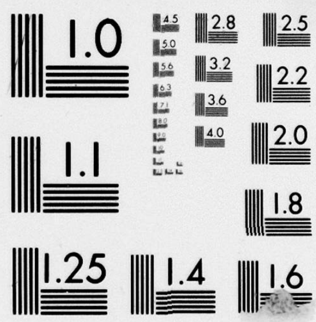

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AFML-TR-79-4092

EXPLORATORY DEVELOPMENT OF DIE MATERIALS FOR ISOTHERMAL FORGING OF TITANIUM ALLOYS

TRW Materials Technology
23555 Euclid Avenue
Cleveland, Ohio 44117

July 1979

TECHNICAL REPORT AFML-TR-79-4092

Final Report for Period 1 May 1976 to 30 April 1979

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This technical report has been reviewed and is approved for publication.

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A program was conducted to develop materials for isothermal forging dies exhibiting higher yield strength and creep resistance than currently used nickel-base superalloy and molybdenum alloy compositions. The technical approach involves two phases, the first including alloy development aimed towards improved casting, powder metallurgy and metastable carbide alloy compositions and the second including the fabrication of tooling of the most promising die material/process for the F-15 trunnion drag brace arresting

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20. Abstract (continued)

gear. The alloy development study included the evaluation of TRW-NASA VIA, TAZ-8A, IN-792 and IN-100 in terms of tensile, creep rupture and thermal fatigue tests as well as lubricant compatibility tests with typical isothermal forging lubricants. The results indicated that a modified TRW-NASA VIA casting composition offered the greatest potential for applicability as a die material. This included a 15% increase in 927°C (1700°F) yield strength, a three fold increase in the creep resistance at 954°C (1750°F)/106.8 MPa (30 Ksi) and improved resistance to typical isothermal forging lubricants compared to the currently used IN-100 die material. For the die fabrication efforts, vacuum induction melting and vacuum casting were used to produce the tooling with the major die cavity impressions cast directly into the inserts. EDM machining was used to produce the finished die impressions. Tooling characterization included tensile and creep-rupture tests on material machined from the castings and indicated significant improvement compared to IN-100 cast in similar section sizes. This included a 35% improvement in yield strength, a three-fold improvement in creep-rupture life and an order of magnitude improvement in time to reach 0.1% creep. The performance of this tooling will be evaluated during the subsequent production of a series of isothermally forged parts under Air Force Contract F33615-76-C-5386, "Isothermal Forging Beta Titanium."

FOREWARD

This Final Technical Report covers all work performed under Contract F33615-76-C-5105 by TRW Inc., Cleveland, Ohio, from 1 May 1976 to 30 April 1979.

This contract was initiated under Project 7351, "Metallic Materials," Task 735108, "Processing of Metals," Work Unit 73510821. The work was performed under the technical direction of Mr. Attwell M. Adair and Mr. William T. O'Hara of the Metals and Ceramics Division, and Mr. Robert J. Ondercin of the Manufacturing Technology Division of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio 45433.

Dr. Charles S. Kortovich of TRW Inc. served as program manager and was responsible for the management and execution of this program. Mr. James M. Marder served as principal investigator. Mr. Donald J. Moracz and Dr. Thomas S. Piwonka assisted in the design and fabrication of the isothermal forging die system resulting from this program.

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I INTRODUCTION

Titanium alloys represent a major materials application for both critical airframe and engine components in advanced high performance aircraft now in service and anticipated for future requirements. These alloys offer material properties, structural stability and efficiency and low life cycle costs unobtainable with other material systems. An important disadvantage associated with titanium alloy components, however, are the high fabrication costs. The energy requirements are high for initial production, alloying and melting, and ingot conversion and contribute significantly to high material costs. In addition, the materials are expensive to fabricate due to the difficulty of working operations. Material utilization is also poor, with the forging-to-finished part weight ratio often being less than 10:1. This need to reduce costs has stimulated the development of sophisticated manufacturing processes directed towards the fabrication of high performance airframe structures and engine components from titanium alloys.

A particularly significant example of this development work is the isothermal forging process. Briefly, isothermal forging is a process in which the dies are heated to the same temperature as the forged material in order to eliminate the die chilling effects. For titanium alloys, this forging temperature is in the range of 871°C (1600°F) to 982°C (1800°F). The major advantages associated with this processing include: (1) improved contour refinement over that normally achieved by conventional forging, (2) increased forging capacity through decreased forging load, (3) reduced numbers of blocking or performing operations and (4) lower costs through reduced material input and elimination of a significant amount of manually controlled surface finishing. The isothermal forging technology as applied to titanium alloys has developed for more than a decade largely through sponsorship of the Air Force Materials Laboratory. As a result, these programs have provided meaningful data on the design of preform shapes and the performance of production size tooling in the stress states and environment experienced in an operating production facility (1-5). The process has advanced to the production stage and typical components include a horizontal stabilizer torque rib (6), a veritical stabilizer center hinge (7), and an engine mount connecting link (7) all for the Air Force F-15 fighter.

In spite of the advantages of isothermal forging, however, it has generally been concluded by each manufacturer involved in the technology that the process can be more cost effective if a more satisfactory die material were available. It is currently estimated that substantial cost savings per part can be realized if a sufficient number of parts can be produced from each die configuration. More specifically, the amortization point for various parts can be reduced significantly if the total cost of the dies can be reduced. Factors which influence the overall economics of isothermal forging die sets include material costs, die cavity sinking and preparation costs and useful lifetime before major reworking or scrapping is required. The currently used die materials include either the molybdenum-base alloys or the nickel-base superalloys. Neither of the materials are completely satisfactory for current isothermal forging die applications. The molybdenum-base alloys require a vacuum chamber or inert gas shield around the die system to prevent oxidation of the dies. While they are very satisfactory for strength level and dimensional stability, their basic cost is high, die cavity production costs are high, and the required oxidation protection equipment is costly. The major problems experienced with the cast

nickel-base superalloy dies used to date has been their inability to retain the desired form under the stress-temperature-time and environment conditions imposed. The cost of the superalloy castings employed for dies and the cost of machining the dies are additional factors which limit the use of superalloy forging dies. It is clear, therefore, that the development of improved die materials would considerably enhance the overall applicability of the isothermal forging process for titanium alloys.

The present investigation was conducted then, to develop materials and processing techniques capable of producing isothermal forging dies having higher strength and increased dimensional stability at a lower overall cost than is possible with present-day nickel-base superalloys and molybdenum alloys. More specifically, the directions and purposes of this program were to determine the applicability of powder metallurgy techniques and appropriate improved casting techniques to the production of die cavity shapes from newly developed superalloy compositions. Target property goals for these candidate die materials included 517 MPa (75 ksi) 0.2% yield strength at 927°C (1700°F) and a time in excess of 30 hours to reach 0.1% plastic creep at 954°C (1750°F)/206.8 MPa (30 ksi). The experimental approach involved two phases. In Phase I - Alloy Development, nickel-base alloy development principles were applied to efforts in which nine powder metallurgy (Task I), nine casting alloy (Task II), and nine metastable carbide alloy (Task III) compositions were evaluated in three series of alloys. Evaluation included mechanical property screening tests on the first two series of alloys and a more complete property evaluation on the two most promising compositions in Series III. The more complete evaluations included tensile, creep-rupture and thermal fatigue tests as well as lubricant compatibility tests with typical isothermal forging lubricant compositions. In Phase II - Die Fabrication, the most promising die material/process combination was used to fabricate tooling for the F-15 trunnion drag brace arresting gear. The performance of this tooling will be evaluated at TRW during the subsequent production of a series of isothermally forged parts under Air Force Contract F33615-76-C-5386, "Isothermal Forging Beta Titanium."

The results of this study are summarized in this final report. It includes a review of the program outline to develop materials and processing techniques capable of producing improved isothermal forging dies, a review of the experimental procedures, a summary of the experimental results, and a discussion of these results.

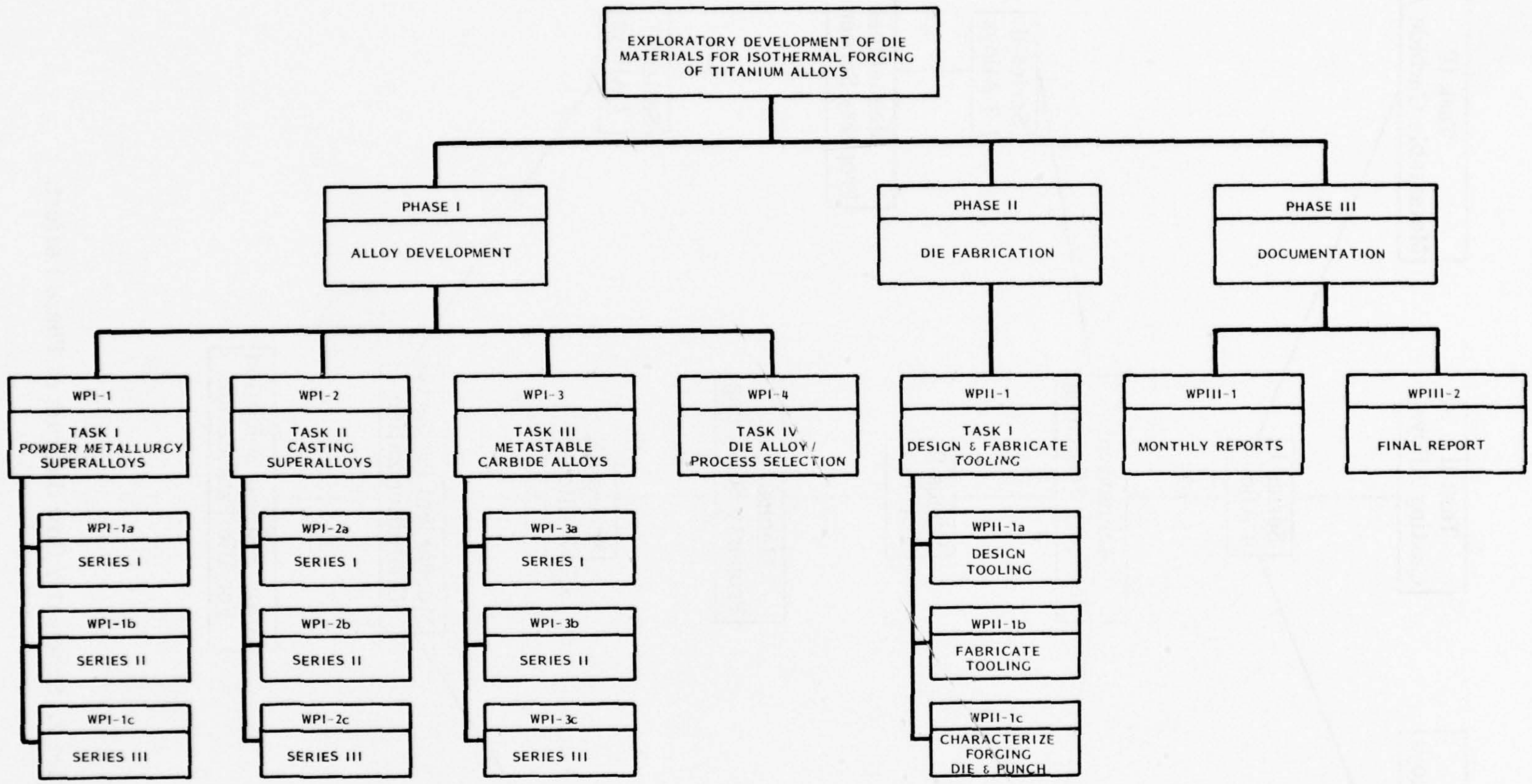


Figure 1. Work Breakdown Structure of program.

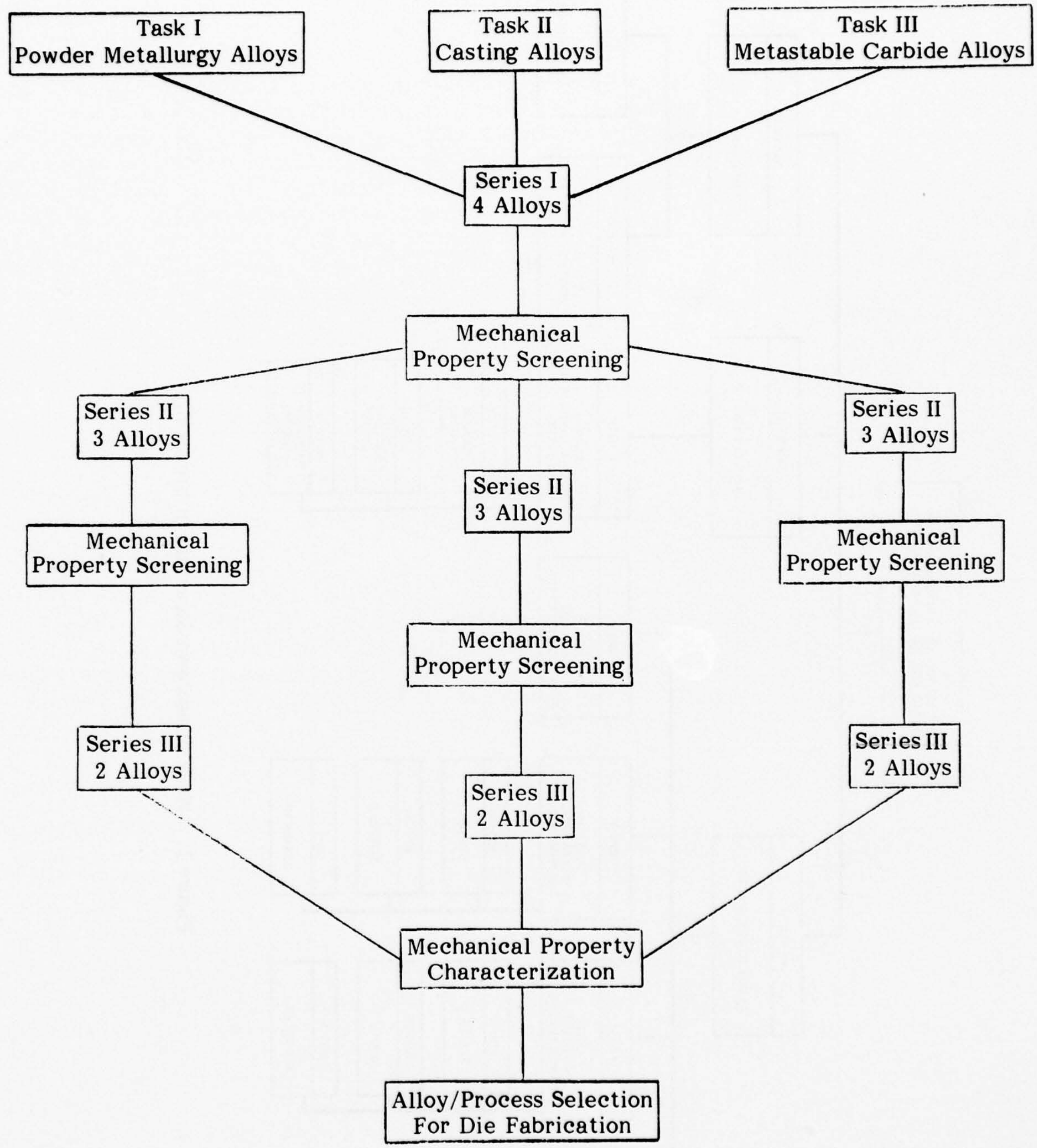


Figure 2. Schematic flow diagram of Phase I effort.

II PROGRAM OUTLINE

The objective of the program was to develop materials and their required processing techniques for the production of isothermal forging dies exhibiting higher yield strength and increased resistance to dimensional distortion at a reduced overall cost than currently possible with present-day nickel-base superalloy and molybdenum alloy compositions. To accomplish this, work was directed towards evaluating the applicability of powder metallurgy techniques and appropriate improved casting techniques for the production of die cavity shapes from new superalloy compositions. The study consisted of two major phases and followed the outline presented in the Work Breakdown Structure of Figure 1. Phase I of the investigation consisted of a three task alloy development effort in which three different alternate die alloy/process methods were studied including powder metallurgy superalloys, cast superalloys and metastable carbide alloys. A fourth task involved die alloy/process selection Phase II consisted of die fabrication of the most promising die material/process combination.

A. Phase I - Alloy Development

The Phase I efforts included evaluation of candidate alloy compositions in order to develop an alloy/process capable of meeting the target properties of the program which included 517 MPa (75 ksi) 0.2% yield strength at 927^{o}C (1700^{o}F) and a time in excess of 30 hours to reach 0.1% plastic creep at 954^{o}C (1750^{o}F)/206.8 MPa (30 ksi). For each of the three tasks, the alloys were divided into three series of compositions. The Series I alloys were selected on the basis of current data to optimize the 927^{o}C (1700^{o}F) tensile and the 954^{o}C (1750^{o}F) creep-rupture resistance. The Series II and III alloys were designed on the basis of the Series I screening studies. The most promising alloy/process combination was selected for the Phase II - Die Fabrication efforts. A schematic flow diagram of the Phase I effort is shown in Figure 2.

1. Task I - Powder Metallurgy Superalloys

The Task I study included the development of nickel-base superalloy compositions compatible with prealloyed metal powder technology. A total of nine different alloys were evaluated in this task, four in Series I, three in Series II and two in Series III.

a. Series I Alloy Studies

The four alloys selected for the Series I work included TRW-NASA VIA, TAZ-8A, IN-792, and IN-100. This selection was made on the basis of the potential each alloy exhibited to achieve program mechanical property goals as well as the anticipated oxidation/sulfidation resistance each would exhibit in the presence of advanced isothermal forging lubricants. The carbon content of these alloys was reduced to 0.05 weight percent to enhance the development of large grain sizes in the consolidated powder metallurgy material. The powder metallurgy alloys were atomized at Homogeneous Metals Inc. from vacuum induction melted ingots produced at the TRW Turbine Components Metals Division. Homogeneous Metals utilized the soluble gas atomization technique employing hydrogen as the soluble gas.

The alloys were compacted by hot isostatic pressing at Industrial Materials Technology. In order to insure complete densification of the powders, a parametric study was conducted on each of the alloys utilizing a cyclindrical can configuration 1.6 cm (5/8 inch) internal diameter by 10 cm (4 inches) long. Once metallographic evaluation had verified that complete densification had occurred, 2 bars each 1.6 cm (5/8 inch) internal diameter by 35.6 cm (14 inches) long were compacted for mechanical property screening studies. Prior to the mechanical property screening studies, however, a heat treatment study was conducted to determine the optimum test microstructure for each alloy. This consisted of grain growth and aging heat treatments to insure an ASTM 2-3 grain size in the material and a discrete particle grain boundary morphology. Mechanical property screening tests consisted of three 927^{o}C (1700^{o}F) tensile tests per alloy and five creep rupture tests per alloy at 954^{o}C (1750^{o}F)/206.8 MPa (30 ksi). The alloy exhibiting the best combination of mechanical properties consistent with the program target goals was selected as the baseline composition for Series II.

b. Series II and III Alloy Studies

On the basis of the Series I results three experimental compositions using TRW-NASA VIA (exhibiting the best combination of properties) as the baseline were formulated in Series II to attain the program target goals. The same powder atomization, compaction (including the HIP parametric study), heat treatment study and mechanical property screening procedures described in Series I were also used in Series II. Based upon these results, the final series of two experimental compositions were formulated for the final alloy optimization of Series III. Again, TRW-NASA VIA was used as the baseline composition.

With the exception of the mechanical property screening study, the same procedures used for Series I and II were used for Series III. At this point a more extensive mechanical property characterization was conducted. Tests included the following for each alloy:

- Five 927^{o}C (1700^{o}F) tensile tests.
- Nine 954^{o}C (1750^{o}F)/206.8 MPa (30 ksi) creep-rupture tests.
- A minimum of two lubricant compatibility tests with OPT112 and Deltaglaze 69 consisting of 80 hours total exposure at a temperature range of 871^{o}C (1600^{o}F) - 982^{o}C (1800^{o}F). Tests were conducted at 55.6^{o}C (100^{o}F) intervals and included metallographic evaluation of the oxidation/sulfidation attack.
- Duplicate thermal fatigue-creep interaction tests conducted on tensile specimens cycled at 927^{o}C (1700^{o}F)/172 MPa (25 ksi) (assuming this as the maximum nominal stress on the die as typical for production isothermal forging practice). Test cycles ran four hours after which specimens were unloaded and cooled for 5-10 minutes and loaded and heated again. Cycles to complete specimen separation were measured.

At the conclusion of this portion of the Phase I effort, the optimum powder metallurgy alloy was selected.

2. Task II - Casting Superalloys

The Task II effort included the development of nickel-base superalloy compositions compatible with current casting technology. Similar to Task I, a total of nine different alloys were evaluated in this task, four in Series I, three in Series II and two in Series III.

a. Series I Alloy Studies

The four alloys selected for the Series I work were identical to the Task I powder metallurgy alloys with the exception that the casting alloys all contained their normal amounts of carbon. The alloys were prepared at the TRW Experimental Foundry in Minerva, Ohio in the form of 22.6 kg (50 pound) 8.9-cm (3.5-inches) x 8.9-cm (3.5-inches) x 17.8-cm (7-inches) long rectangular blocks. One block was made of each of the alloys using current vacuum precision investment casting techniques. Mechanical property screening studies identical to those described in Series I of Task I were conducted on specimens machined from the corner portions of the cast die blocks. This represents the situation at the surface of the isothermal forging die. As a check, tests were also conducted on specimens machined from the center portion of the block to determine section size effects. All specimens were tested in the as-cast condition and the alloy with the best combination of mechanical properties consistent with the program target goals was chosen as the baseline composition for Series II.

b. Series II and III Alloy Studies

On the basis of the Series I results, three experimental compositions using TRW-NASA VIA (exhibiting the best combination of properties) as the baseline were formulated in Series II to attain the program target goals. The same casting configuration and mechanical property screening procedures employed in Series I were also utilized for Series II. Based upon these results, the final series of two experimental compositions was formulated for the final alloy optimization of Series III. Again, TRW-NASA VIA was used as the baseline composition. The Series III alloys were subjected to the same extensive mechanical property characterization described in Task I, Series III for the powder metallurgy alloys. This necessitated the production of two large cast blocks per alloy composition. At the conclusion of this effort the optimum cast superalloy composition was identified.

3. Task III - Metastable Carbide Alloys

The Task III efforts included an evaluation of the potential of metastable carbide powder metallurgy alloys to satisfy the need for improved isothermal forging die materials. Metastable carbide processing was designed to overcome the problem of the presence of stable carbides in the grain boundary regions of superalloy powder compacts which prevented the important grain growth necessary for good high temperature stress rupture properties. In this technique, developed at TRW under NASA (8) and Naval Air Systems Command (9) sponsorship, carbon is selectively added in the form of metastable carbides (e.g., Cr_2C_3 SiC, VC) to a carbon-free prealloyed base composition. After consolidation, high temperature heat treatments are applied which solution the metastable

carbides, allowing grain growth to occur and releasing carbon for subsequent precipitation as discrete particle grain boundary carbides. A total of nine different metastable carbide alloys were evaluated in this investigation, with four being studied in Series I, three in Series II and two in Series III.

a. Series I Alloy Studies

The alloys selected for the Series I work were identical to the powder metallurgy and casting alloys with one important exception. The carbon in these alloys was added in the form of metastable SiC. SiC was selected for the present program as a result of the difficulty in the previous Naval Air Systems Command sponsored program conducted at TRW (9) to completely solution metastable VC and obtain the desired ASTM 2-3 grain size. In addition to this, studies conducted on the effect of Si additions to superalloy compositions indicated that increased oxidation resistance was obtained with small amounts of Si additions (10).

The carbon-free powder master alloys were produced at Homogeneous Metals, Inc. from vacuum induction melted ingots produced by TRW. The soluble gas atomization technique was employed with hydrogen as the soluble gas. Attrition was used to produce a homogeneous distribution of SiC particles throughout the powder mixture. The proper amounts of these additions were attrited with the prealloyed master alloy powder in the TRW 10-S Szegvari Attritor, under an argon atmosphere, to produce the desired alloy composition. The powders were compacted by hot isostatic pressing at Industrial Materials Technology using can configurations and procedures described previously for the powder metallurgy alloys. Similar to the Task I work, a parametric study was conducted to insure complete densification of the powders.

Prior to the mechanical property screening evaluations, a heat treatment study was conducted to develop the optimum grain structure with a discrete particle carbide morphology in the grain boundaries for each metastable carbide alloy. The heat treatment study involved three steps, a high temperature grain coarsening anneal, a partial solutioning treatment and a low temperature aging treatment. The grain coarsening anneal was conducted to accomplish two objectives: (1) to solution the metastable carbide thus freeing carbon for subsequent precipitation, and (2) to develop the desired grain size and morphology. Mechanical property screening studies were then conducted on material given the optimized heat treatment according to the procedures described previously for Task I and II.

b. Series II and III Alloy Studies

On the basis of the Series I results, three experimental compositions using TRW-NASA VIA (exhibiting the best combination of mechanical properties) as the baseline were formulated for further alloy optimization in Series II. The same powder atomization, attrition, compaction, heat treatment and mechanical property screening efforts described in Series I were also used in Series II. Based upon these results, a final series of two experimental compositions were formulated for the alloy optimization of Series III. Again, TRW-NASA VIA was used as the baseline composition. The Series III alloys were then subjected to extensive mechanical property characterization. At the conclusion of this task the optimum metastable carbide powder metallurgy alloy was identified.

4. Task IV - Die Alloy/Process Selection

The Task IV efforts included analysis of the mechanical property characterization data generated in Tasks I, II and III and selection of a single die material/fabrication method for the die fabrication efforts of Phase II. A modification of cast TRW-NASA VIA was selected for the Phase II work.

B. Phase II - Die Fabrication

The Phase II efforts involved the fabrication of isothermal forging tooling for evaluation of the single die material/fabrication process selected during the Phase I alloy development work. A candidate forging component was selected in Phase II the evaluation of which will be co-ordinated with Air Force Contract F33615-76-C-5386, "Isothermal Forging of Beta Titanium." In addition to component selection, Phase II work included tooling design and fabrication as well as characterization of the isothermal forging dies.

1. Forging Component Selection

The component to be forged was selected from current requirements for aircraft structural or engine parts having a plan area of 645 cm^2 (100 square inches). Since the F-15 and F-16 aircraft and the F-100, the TF-34 and F-101 engine all are of current interest to the Air Force, the requirements for these engines were all considered in making recommendations for the part to be forged. For the program a static critical component for the F-15 aircraft was selected. The part is referred to as a trunnion and is currently machined from a conventional oversize die forging procured in annealed Ti 6Al-5V-2Sn. This component contains extensive thin web areas, reinforcing ribs, and a slightly heavier section for an arresting gear drag brace attachment.

2. Tooling Design

The tooling design activity was directed to provide as thin an envelope on the part configuration as possible consistent with stock removal requirements to assure the elimination of any alpha case or surface contamination and for minimum draft angles and radii consistent with good forging die design. The flash pad design, an important feature for controlling the flow of the workpiece material and the anticipated loading on the dies was engineered to develop a 172 MPa (25 ksi) nominal stress on the dies. The TRW advanced forging die design system was used for calculating the loading on the dies during the design stage. The design efforts also incorporated the preform design since the die loading is also determined by the metal distribution in the preform or blocker shape.

3. Tooling Fabrication

In this effort, the die alloy/fabrication process selected in Phase I was used to fabricate the isothermal forging tooling. This tooling consisted of a forging die and punch assembly. Since a casting modification of TRW-NASA VIA was selected, vacuum induction melting and vacuum casting were used to produce the tooling. The ceramic casting molds were produced by Cast Masters, Inc. of Bowling Green, Ohio and shipped to the Cannon Muskegon Company, of Muskegon, Michigan, where they were vacuum cast

from master metal of the desired composition, also produced by Cannon Muskegon. The castings were shipped to Contoured Electrode of Mentor, Ohio for final finish machining operations.

4. Tooling Characterization

The forging die and punch assembly was characterized by visual observation, photography, dimensional inspection of selected critical areas and fluorescent penetrant inspection to enable a complete documentation of the process history of the tooling assembly. In addition, mechanical property evaluations were conducted on specimens machined from the castings to characterize this material in comparison to the compositions tested during the alloy development efforts of Phase I. Testing included 927°C (1700°F) tensile tests and 954°C (1750°F)/206.8 MPa (30 ksi) creep rupture tests as well as a metallographic analysis to characterize the casting microstructure. Additional tensile and creep rupture testing was also conducted on two other materials for further comparisons. These included air-melted and cast modified TRW-NASA VIA of similar composition to the forge tooling and air-melted and cast IN-100 superalloy. These tests were conducted to (1) identify any mechanical property performance differences between air-melted and cast VIA type material and vacuum processed alloy, and (2) to establish mechanical property differences between the modified TRW-NASA VIA and the superalloy material currently in use as an isothermal forging die material.

C. Environmental Impact

The improved TRW-NASA VIA isothermal forging die superalloy resulting from this program can be produced using currently installed equipment in a number of organizations throughout the casting industry. The currently used IN-100 superalloy die material is manufactured on equipment of this type and no unfavorable environmental problems will thus be created by employing the new TRW-NASA VIA composition. The current plants and equipment conform to local, state, and federal regulations regarding protection of the environment.

III PHASE I - ALLOY DEVELOPMENT

A. Task I - Powder Metallurgy Superalloys

The Task I efforts included evaluation of nine candidate alloy compositions compatible with prealloyed powder technology. The nine alloys were divided into three series with four alloys in Series I, three in Series II and two in Series III. The following sections contain descriptions of the procedures used for the development of these powder metallurgy nickel-base superalloys as well as a discussion of the results of the alloy development effort.

1. Experimental Procedures

a. Powder Preparation

Hydrogen gas atomization was used to produce the powder metallurgy alloys for this program. For this operation, 22.6 kg (50 pound) vacuum induction melted ingots of the desired compositions were first produced at the TRW Turbine Components Metals Division and then shipped to Homogeneous Metals, Inc., of Herkimer, New York for atomization in their 22.6 kg (50 pound) pilot facility. There, the ingots of superalloy material were vacuum melted to insure proper mixing and were then supersaturated with hydrogen gas under high pressure. The liquid metal was introduced to a vacuum chamber through an orifice, where the pressurized liquid erupted into particles. Particle size distribution was relatively broad, ranging from -20 mesh to below -325 mesh. Particle size control was achieved by selecting the proper orifice size and the pressure differential. A yield of 55-65% of -80 mesh powder was obtained during the atomization operations. The inherent advantage of this powder production method was the fact that thorough alloy mixing took place during melting, that no entrapped gases remained in the powder particles, and that consistently low oxygen content powders (<100 ppm) were obtained. Subsequent to atomization the powders were stored in argon to minimize contamination.

b. Powder Consolidation

The powder alloys were compacted by hot isostatic pressing by Industrial Materials Technology of Woburn, Massachusetts. In order to insure complete densification of the powders, a parametric study was conducted on each of the alloys utilizing a cylindrical can configuration 1.6 cm (5/8 inch) internal diameter by 10 cm (4 inches) long. Can material consisted of 0.3-cm (1/8-inch) thick mild steel and vacuum outgassing at $540^{o}C$ ($1000^{o}F$) for 24 hours was conducted prior to final can sealing to drive off argon gas adsorbed on the powder surfaces. These adsorbed gases can result in gas porosity during subsequent high temperature heat treatments of superalloy powders (11). Once complete densification was verified by metallographic evaluation, the optimized hot isostatic pressing parameters were used to produce 2 bars each 1.6 cm (5/8 inch) internal diameter by 35.6 cm (14 inches) long for mechanical property screening studies for the Series I and II alloys. Four such bars were prepared for the more extensive mechanical property evaluations on the Series III compositions.

c. Mechanical Property Screening Studies (Series I and II Alloys)

Prior to the mechanical property screening evaluations, a heat treatment study was conducted to determine the optimum test microstructure for each alloy. This consisted of grain growth and aging heat treatments to produce an ASTM 2-3 grain size in the material and a discrete particle grain boundary morphology. For the four alloys of Series I and the three alloys of Series II the mechanical property screening tests consisted of three 927°C (1700°F) tensile tests per alloy and five creep rupture tests per alloy at 954°C (1750°F). The test specimen configuration for all mechanical property tests is shown in Figure 3.

(1) Tensile Tests

Tensile testing was performed in air at 927°C (1700°F). All tests were carried out on an Instron Universal Test machine with a crosshead speed of 0.012 cm/minute (0.005 in/minute) until yielding occurred. Temperature was controlled to within 2°C (3°F). Ultimate tensile strength, 0.2% offset yield strength, percent reduction of area and percent elongation were recorded.

(2) Creep-Rupture Tests

Creep-rupture tests were performed in air at 954°C (1750°F)/206.8 MPa (30 ksi). Tests were conducted in Satec constant load, lever arm units and temperature was maintained with a 2°C (3°F) tolerance. Failure time, time to 0.1% creep, percent reduction of area and percent elongation were recorded.

d. Mechanical Property Characterization (Series III Alloys)

An extensive mechanical property characterization was conducted on the two Series III alloys. These evaluations included five tensile tests, nine creep-rupture tests, lubricant compatibility tests and thermal fatigue-creep interaction tests. The procedures for the tensile and the creep-rupture tests were identical to those for the screening studies. The procedures for the lubricant compatibility and the thermal fatigue-creep interaction tests are described in the following sections.

(1) Lubricant Compatibility Tests

Lubricant compatibility tests were conducted with OPT 112 and Deltaglaze 69 lubricant compositions. These lubricants were selected as being representative of advanced state-of-the-art lubricant technology (12) compatible with advanced isothermal forging die systems. OPT 112 is an experimental lubricant developed and patented by TRW. It has a composition comprised of 14% by weight fine boron nitride powder in a vitreous phase of 67% diboron trioxide and 33% silica glass frit with a transition metal oxide addition. Deltaglaze 69 is a commercial product available from Acheson Colloids Co. Deltaglaze 69 is an aluminoborosilicate glass; however, the composition is considered proprietary by Acheson. The testing consisted of 80 hours total exposure at a temperature range of 872°C (1600°F) - 982°C (1800°F). The tests were conducted at 55.6°C (100°F) intervals and included metallographic evaluation of the oxidation/sulfidation attack produced on 1.3-cm (1/2-inch) diameter specimens coated with the lubricant and exposed to temperature in an air atmosphere furnace. All lubricant compatibility results were compared to test results obtained on standard cast IN-100 material currently used in isothermal forging die systems.

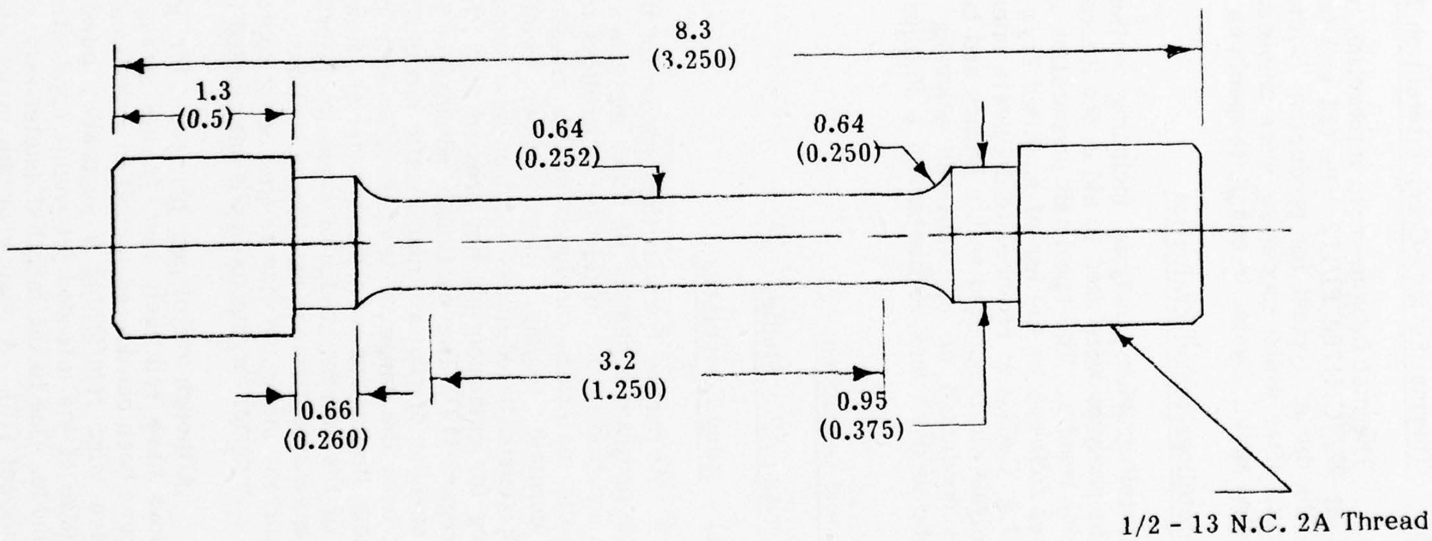


Figure 3. Schematic illustration of test specimen configuration used for all mechanical property characterization. Dimensions in inches are shown in parentheses.

(2) Thermal Fatigue-Creep Interaction Tests

Thermal fatigue-creep interaction tests were conducted on tensile specimens cycled at 927°C (1700°F)/172 MPa (25 ksi) (assuming this as the maximum nominal stress on the die as typical for production isothermal forging practice). Test cycles ran four hours after which specimens were unloaded and cooled for 5-10 minutes and loaded and heated again. Cycles to complete specimen separation were recorded.

e. Metallographical Evaluation

Metallographic analysis including optical metallographic, SEM and electron microprobe analyses were used to aid in the interpretation of the processing and mechanical property results. The specimen preparation included standard grinding and polishing procedures followed by the use of modified Fry's etch (150 ml H_2O, 50 ml HCl, 25 ml HNO_3 and 1 g $CuCl_2$) as required to delineate microstructural features. Optical metallographic analysis was conducted with a Bausch and Lomb Research II metallograph, SEM analysis was conducted on an AMR-900 scanning electron microscope and the electron microprobe analysis was conducted on a Phillips AMR/3 electron microprobe analyzer.

2. Results and Discussion

a. Series I Alloy Studies

(1) Alloy Formulation

As part of the selection process for the first series of alloys for the various tasks of this program a number of other materials and/or processing techniques were considered in addition to the nickel-base superalloys to satisfy the need for superior isothermal forging die materials. Included in this assessment of material and process feasibility were ceramic tooling, tungsten-fiber reinforced superalloys, cladding techniques for molybdenum based alloys and iron base superalloys. Ceramics have been evaluated as tooling for extrusion and hot pressed Si_3N_4 has been reported to be most promising in this regard (13). The particular advantages associated with this material appear to be high modulus of rupture, high elastic modulus, good resistance to oxidation and thermal shock over the range of temperatures anticipated for isothermal forging. There are two serious limitations with these materials, however. First is the problem of die sinking. Many of the current grades of ceramic materials cannot be EDM machined and would require expensive diamond grinding operations. A second disadvantage is size availability, with current maximum size ranges being approximately 20.3-25.4 cm (8-10 inches) (14). Most isothermal forging tooling is anticipated to be much larger in size.

Although developed primarily for jet engine applications, fiber reinforced superalloys have relatively high tensile strength and stability at elevated temperatures and have been considered for use in isothermal die applications particularly in terms of tungsten wire reinforcing a superalloy powder hot isostatically pressed compact (15). Because of the intended jet engine application, most reported testing has been in the tensile mode. Due to the length-diameter ratio of the fibers, the compressive behavior may be suspect (15). A review of the major attributes of this system (high tensile strength and low coefficient of thermal expansion) suggests that fiber-reinforcement would probably be most useful in an insert holder type application.

However, continuous winding of a large holder would require very long monolayer tapes which are currently not available. Since current airframe structural parts require alpha beta finish forging, the potential benefits of fiber-reinforcing are reduced compared to the nickel-base superalloys.

Although molybdenum-base alloys are currently used in certain isothermal forging die applications (16,17), a serious limitation to their extended application is the poor oxidation resistance of these materials. This necessitates the use of expensive inert atmosphere forging environments such as vacuum or argon gas to protect the dies. For example, in certain applications molybdenum disilicide surface treatments have been used to offer protection but these would be totally inadequate under the loading stresses anticipated in isothermal forging operations. The glass industry employs platinum coatings to protect injection molds made from molybdenum-base alloys, but these coatings would be cost prohibitive if applied to isothermal forging dies. As a result of these considerations it was concluded that cladding techniques for molybdenum-base alloys do not have sufficient feasibility to be applied to isothermal forging dies.

There is some consideration being given to the use of iron-base superalloys for use at the lower end of the temperature range for isothermal forging die applications. Because of their strength limitations, however, the development of iron-base alloys for use at and above 649°C (1200°F) has not been as successful as that for cobalt or nickel-base alloys. Stainless steels and their modifications still find wide applications at temperatures in excess of 649°C (1200°F) because of their outstanding oxidation resistance. However, where stresses are high, about 103.4 MPa (15 ksi) or more, iron-base alloys usually cannot be considered (18). These alloys are commonly employed for automotive valve applications (19), gas turbine engine rotors, bolts and sheet parts, but yield strengths at 760°C (1400°F) are all below 344.7 MPa (50 ksi) (20). From these strength considerations, then, the iron-base superalloys do not exhibit sufficient potential to perform as adequate isothermal forging die materials.

Recent work conducted on compacted advanced nickel-base superalloy prealloyed powders has generally been focused on turbine disk applications where test data were limited to 816°C (1500°F) maximum (21,22) or on blade applications where these tests were usually conducted at or above 982°C (1800°F) (8,23). These results, in conjunction with work conducted on hot isostatically pressed IN-100 (24). and other nickel-base superalloys (25,26), do, however, suggest direction for the application of prealloyed powders for the 816°C (1500°F) - 1038°C (1900°F) temperature range of interest for isothermal forging dies. Of primary importance here is the development of an ASTM 2-3 grain size in the powder metallurgy product (27). This, coupled with proper aging heat treatments to develop a discrete particle grain boundary morphology offers potential for increased tensile strength, creep resistance and thermal fatigue resistance in forging dies. Data from the powder metallurgy studies conducted to date suggest that lower carbon contents can be used effectively to obtain larger grain size in powder metallurgy superalloys (28). This results from the fact that fewer stable carbides are present in the matrix to pin grain boundaries thus preventing effective grain growth.

As a result of these considerations as well as those concerning the isothermal forging die program goals, four nickel-base superalloys were selected for the Series I powder metallurgy work. Efforts on these alloys were directed toward establishing a baseline composition from which alloy development could proceed in Series II and III. The alloys included TRW-NASA VIA, TAZ-8A, IN-792 and IN-100. For each of these powder metallurgy alloys the carbon content was reduced to 0.05% to enhance the development of large grain sizes in the consolidated material. TRW-NASA VIA was selected because it has about the highest high temperature strength properties of present-day conventional (i.e., non directionally solidified, single crystal, eutectic or oxide dispersion strengthened) nickel-base alloys. It also appears to have good corrosion and thermal fatigue resistance (29). The original TRW-NASA VIA contained up to 0.5% rhenium. However, work conducted under TRW internal sponsorship (30) indicated that rhenium is not essential for achieving optimum mechanical properties. Because of this and the relatively high cost of rhenium, it was eliminated from the Series I VIA composition. TAZ-8A was selected because of its reported (31) good high temperature strength properties as well as its superior oxidation resistance compared with alloys such as VIA and IN-100. It also appears to have excellent thermal fatigue resistance (31). IN-792 was chosen because it has high temperature strength comparable to IN-100, the current standard cast nickel-base die material, but its corrosion resistance, especially its hot corrosion resistance, is significantly better (32). IN-100 was selected because it is the current nickel-base superalloy die material used most extensivley for isothermal forging applications and thus represents a baseline for comparison purposes.

The aim compositions of the four Series I alloys are listed in Table I along with the actual compositions in weight percent of material processed during the HIP parametric study. Specimens of this particular material were used for the mechanical property screening evaluations. For all of the powder metallurgy alloys the carbon content was reduced from the normal levels to enhance the development of large grain sizes in the consolidated powder metallurgy material. Comparison of the aim compositions with the actual chemistries indicated that, in general, the desired compositions were achieved. For the IN-100 alloy, however, the chromium and the cobalt contents were above the intended levels. This resulted from the fact that powder of this particular composition was already available at Homogeneous Metals, Inc., and was used for this program. This was justified in view of the similarity of the composition to the IN-100 intended for use in Series I. All of the oxygen levels were considered acceptable in view of recent results obtained on advanced turbine disk alloys in that appreciable mechanical property degradation would not occur until above 200 ppm oxygen (33).

(2) <u>HIP Parametric Studies</u>

The HIP parametric study was conducted on the Series I alloys to determine the processing conditions required to achieve complete densification in the consolidated powders. The initial processing of alloys TRW-NASA VIA, TAZ-8A and IN-792 was conducted at 1204°C (2200°F)/103.4 MPa (15 ksi) for four hours and resulted in complete densification in the compacts. The resultant grain size, however, was extremely fine (approximately ASTM 7-8) for all three alloys. Subsequent to compaction, grain growth heat treatments were investigated to develop a larger grain size in the structures. These studies indicated that grain growth could be achieved in these alloys by exposures up to 1316°C (2400°F) for 4-6 hours. Although grain sizes in excess of ASTM 3 were

Table I

Task I (Powder Metallurgy Superalloys) Series I Alloy Compositions[1]

Alloy		C[2]	C	Cr	Mo	Ti	Al	Co	W	Zr	B	Ta	Cb	Hf	O_2	N_2	Other
TRW-NASA VIA	Aim	0.13	0.05	6.1	2.0	1.0	5.4	7.5	5.8	0.13	0.02	9.0	0.5	0.43			
	Actual		0.034	5.63	2.01	.88	5.29	7.73	6.01	0.06	0.016	9.38	0.65	0.51	80	14	
TAZ-8A	Aim	0.13	0.05	6.0	4.0		6.0		4.0	1.0	0.004	8.0	2.5				
	Actual		0.08	5.96	3.88		5.87		4.42	1.01	0.005	8.37	2.61		163	63	
IN-792	Aim	0.12	0.05	12.4	1.9	4.5	3.1	9.0	3.8	0.10	0.02	3.9					
	Actual		0.052	12.25	1.88	4.15	3.05	9.02	3.39	0.053	0.028	3.65			133	37	
IN-100	Aim	0.18	0.05	10.0	3.0	4.7	5.5	15.0		0.06	0.014						1.0V
	Actual		0.082	12.45	3.23	4.29	5.03	18.33		0.06	0.023				127	45	0.73V

(1) In weight percent, except for oxygen and nitrogen which are reported in parts per million.

(2) These are the normal carbon contents for these alloys as produced in the cast form. Carbon has been lowered for the powder metallurgy study to develop large grain size (ASTM 2-3) in the consolidated powder.

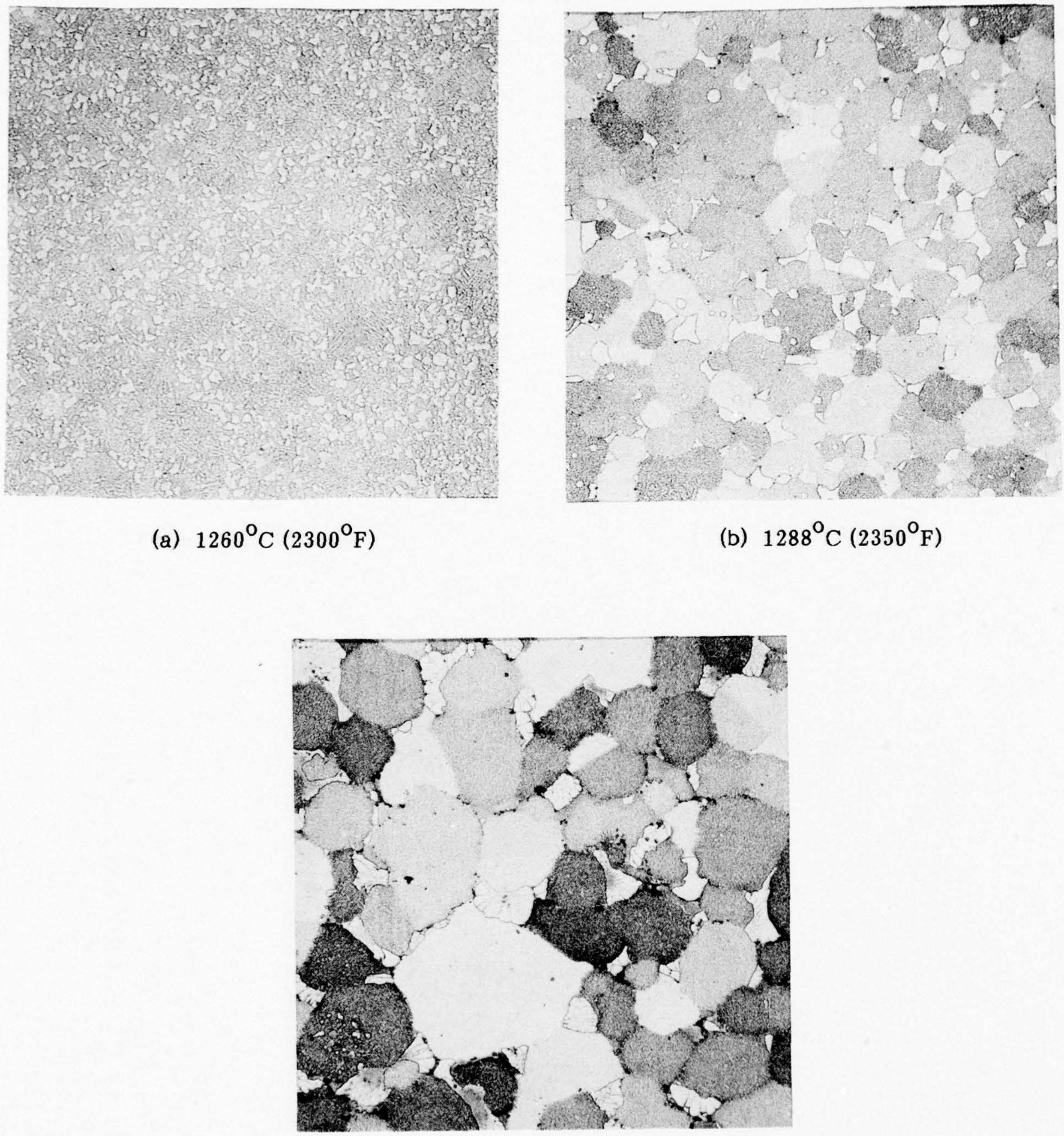
produced in all three alloys, the heat treatments resulted in porosity throughout the microstructures, the result of localized incipient melting. The HIP parametric study was therefore expanded to include higher consolidation temperatures in an effort to eliminate porosity while achieving larger grain sizes. Previous studies employing consolidation temperatures approaching incipient melting have resulted in appreciable grain growth in powder metallurgy superalloys (34,35)

HIP consolidation temperatures of 1260°C (2300°F), 1288°C (2350°F) and 1316°C (2400°F) were employed for this study. The following data indicate the resulting ASTM grain size variations observed as a function of HIP temperature:

	1204°C (2200°F)	1260°C (2300°F)	1288°C (2350°F)	1316°C (2400°F)
TRW-NASA VIA	7-8	6-7	4-6	2-4
TAZ-8A	7-8	7-8	4-5	3-5
IN-792	7-8	7-8	2-3	1

The strongest response in grain size to the higher temperature HIP treatments was found in IN-792, in which an ASTM 1 grain size was observed. The TRW-NASA VIA had an ASTM 2-4 grain size after the 1316°C (2400°F) HIP operation, indicating a substantial improvement over the grain size achieved at lower HIP temperatures. The TAZ-8A alloy evidenced a grain size increase through the use of increased HIP temperatures, but also showed a greater degree of incipient melting at 1316°C (2400°F) as opposed to 1288°C (2350°F). Examples of the changes in the various microstructures and grain sizes as a function of HIP temperature are shown in Figures 4, 5, and 6 for TRW-NASA VIA, TAZ-8A and IN-792, respectively. Note the complete absence of any remnants of the as-solidified dendritic structures in the powders with increasing HIP temperature. Grain size considerations resulted in the selection of a 1316°C (2400°F) HIP temperature for TRW-NASA VIA and IN-792, which exhibited ASTM 2-4 and ASTM 1 grain sizes respectively after the HIP treatment. A HIP temperature of 1288°C (2350°F) was selected for TAZ-8A, which resulted in a grain size of ASTM 3-5. Higher HIP temperatures (as determined by heat treatment exposures at higher temperatures) for these alloys resulted in unacceptably high levels of incipient melting throughout the microstructure. An example of a high level of incipient melting is shown in Figure 5a for the TAZ-8A alloy.

The HIP parametric study for the IN-100 alloy followed along somewhat different lines. The initial HIP operation at 1204°C (2200°F) resulted in a heavy decoration of titanium rich carbides along the surfaces of the powder particles during consolidation. This type of precipitation has been observed previously in IN-100 (36) as well as other superalloys such as Rene' 80 (37), Astroloy (38) and IN-713LC (39) and has been termed prior particle boundary carbide formation. The carbides form as the result of dissolution of finely dispersed MC carbide particles within the superalloy powders during the heat up before consolidation and subsequent reprecipitation on the powder surfaces. Their presence has been shown to be deleterious for two reasons. First, grain coarsening is retarded because grain boundaries cannot move past the carbide network. Second, mechanical properties are degraded because in many cases the almost continuous films of the brittle carbide phases have led to premature failure which is characterized by separation along the prior particle boundaries. Alloy modification has been the most



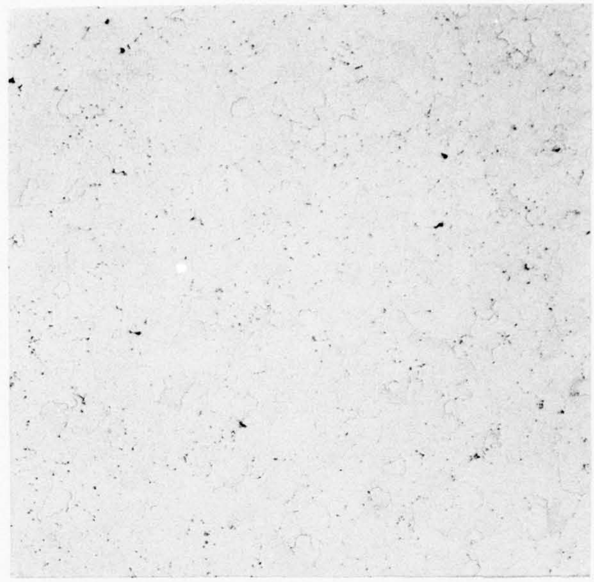
(a) 1260°C (2300°F)

(b) 1288°C (2350°F)

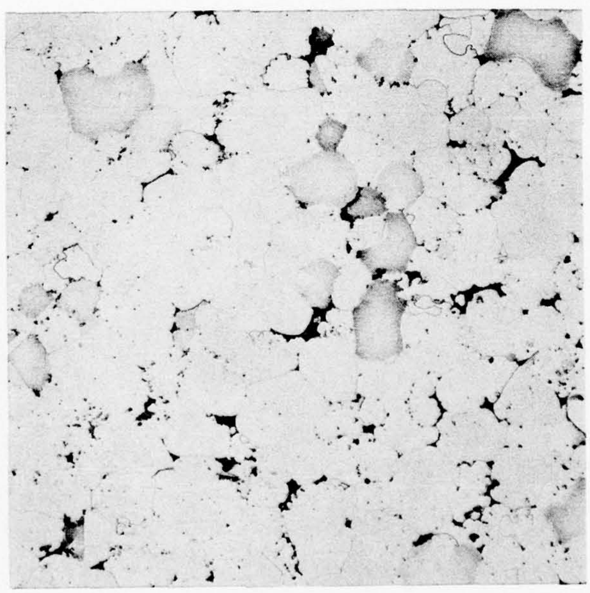
(c) 1316°C (2400°F)

Figure 4. Light photomicrographs of Task I (powder metallurgy) Series I TRW-NASA VIA alloy after HIP at various temperatures for 4 hours and 103.4 MPa (15 ksi). Magnification - 100X

(a) 1260°C (2300°F)

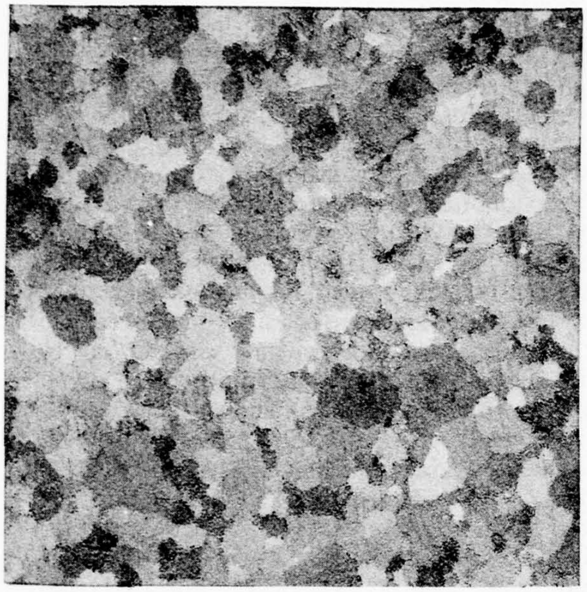


(b) 1288°C (2350°F)

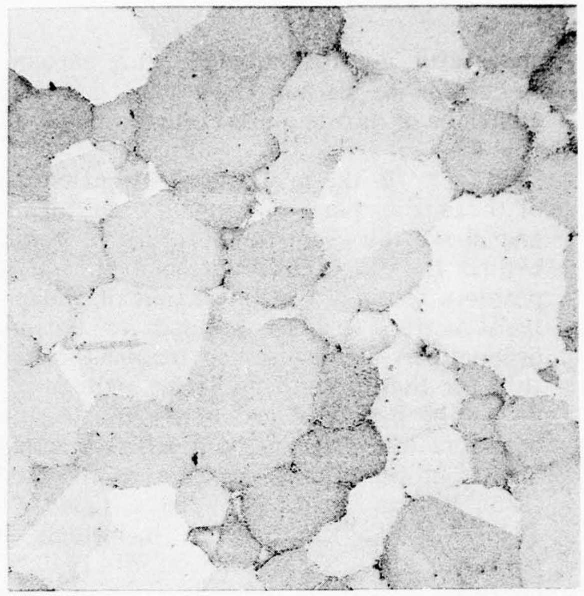


(c) 1316°C (2400°F)

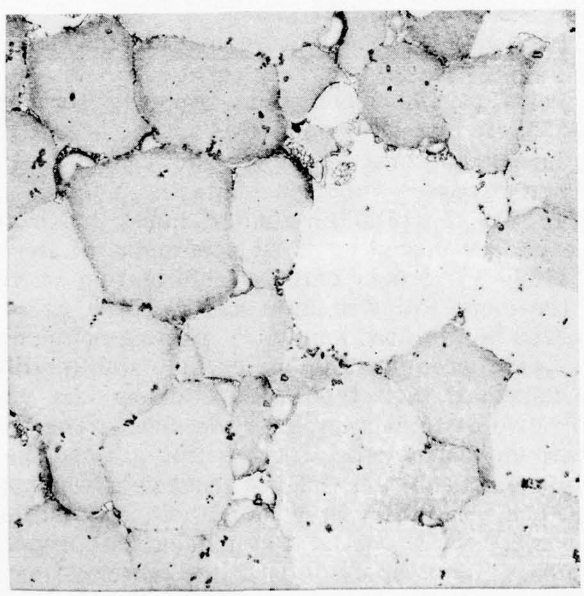
Figure 5. Light photomicrographs of Task I (powder metallurgy) Series I TAZ-8A alloy after HIP at various temperatures for 4 hours and 103.4 MPa (15 ksi). Magnification, 100X.



(a) 1260°C (2300°F)



(b) 1288°C (2350°F)



(c) 1316°C (2400°F)

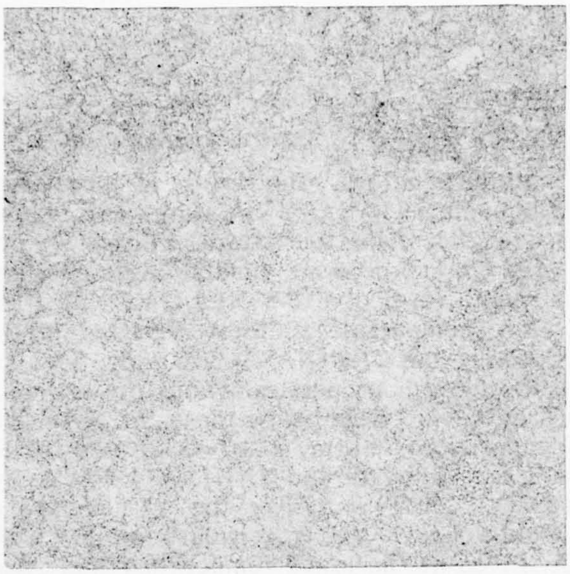
Figure 6. Light photomicrographs of Task I (powder metallurgy) Series I IN-792 alloy after HIP at various temperatures for 4 hours and 103.4 MPa (15 ksi). Magnification - 100X

successful method to avoid the carbide precipitation along prior particle boundaries. Reduction of carbon from 0.18 and 0.15 weight percent to 0.06 weight percent and additions of hafnium and columbium have been most successful in this regard (38,40).

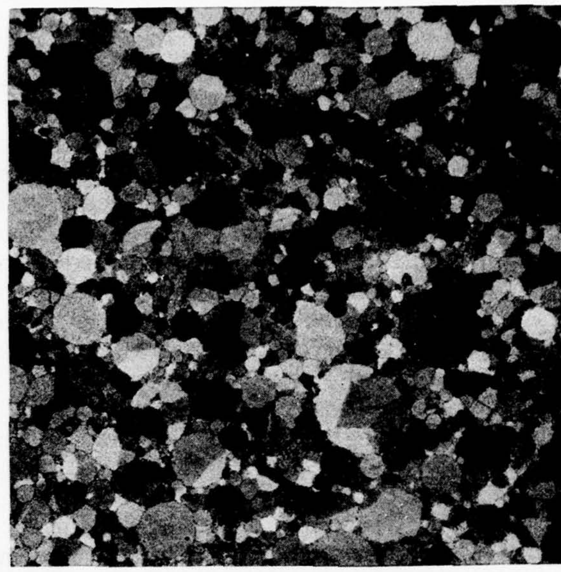
In the present investigation efforts were directed at minimizing the formation of these prior particle boundary carbides by HIP consolidation at temperatures both below and above the usual temperature ranges in which the carbides precipitate. The reasoning behind the HIP consolidation at the lower temperature was to completely densify the powders without the formation of the prior particle boundary carbides after which heat treatments could be applied to optimize the microstructure for high temperature properties. The reasoning behind the high HIP consolidation temperature was similar to that for the other three Series I alloys in that densification as well as grain growth could be achieved by HIP just below the incipient melting temperature. As shown in Figure 7, both the low and the high HIP consolidation temperature were successful for IN-100. Shown in this figure are the as HIP'ed microstructures for powders HIP'ed at 1010°C (1850°F) Figure 7a, and 1288°C (2350°F), Figure 7c, in comparison to powder HIP'ed at 1245°C (2275°F), Figure 7b, which exhibited considerable prior particle carbide precipitation. Consolidation at 1010°C (1850°F) resulted in considerably less prior particle boundary carbide precipitation than the 1245°C (2275°F) consolidation temperature. The 1288°C (2350°F) HIP operation resulted in appreciably larger grain size, approximately ASTM 1-2, which compared favorably with that shown for IN-792 in Figure 6a. On the basis of the reduced prior particle boundary carbide formation and the large grain size, both the 1010°C (1850°F) and the 1288°C (2350°F) HIP consolidation temperatures were selected for the mechanical property screening evaluation on IN-100.

(3) Mechanical Property Screening Evaluation

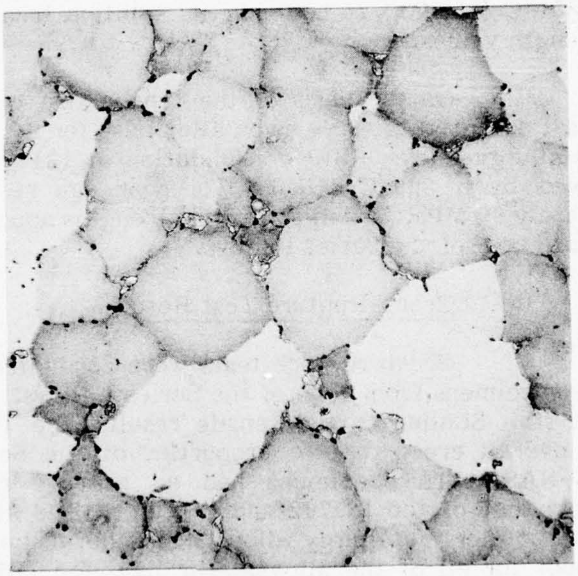
Prior to the mechanical property screening evaluations a heat treatment study was conducted to determine the optimum test microstructure for each alloy. Because the gamma-prime precipitates in the TRW-NASA VIA and TAZ-8A alloys could not be completely solutioned without encountering incipient melting, the alloys were simply heat treated at 899°C (1650°F) for 24 hours to promote precipitation and stabilization of grain boundary carbides. Test specimens of IN-792 were prepared with either a simple 899°C (1650°F) 10 hour carbide stabilization or with a 1121°C (2050°F) 2 hour partial solution treatment followed by a 871°C (1600°F) 24 hour age. The partial solution treatment resulted in a limited amount of gamma-prime coarsening, while the age resulted in fine gamma-prime precipitation and carbide stabilization. This heat treatment was intended to give improved high temperature creep and yield strength properties through the presence of two distinct gamma-prime sizes. The IN-100 was also tested in two different heat treatment conditions. For the fine grained material HIP consolidated at 1010°C (1850°F) a standard solution and age treatment was applied because sufficient grain growth increases could not be achieved without substantial amounts of incipient melting at the grain boundary triple points. The mechanical properties of this fine grained material thus offered a base for comparison with the larger grained material consolidated at 1288°C (2350°F). The standard solution and age treatment included 1204°C (2200°F)/4 hours/air cool + 1080°C (1975°F)/4 hours air cool + 871°C (1600°F)/16 hours/air cool + 760°C (1400°F)/24 hours/air cool. The 1204°C (2200°F) step is a gamma-prime solution treatment. The 1080°C (1975°F) step resulted in gamma-prime precipitation while the 871°C (1600°F) step produced a discrete particle grain boundary carbide morphology. The 760°C (1400°F) treatment resulted in additional fine gamma-prime precipitation. Similar to the TRW-NASA VIA and TAZ-8A HIP consolidated at high temperature, the IN-100 consolidated at 1288°C (2350°F) was aged for 24 hours at 899°C (1650°F).



(a) 1010°C (1850°F)



(b) 1245°C (2275°F)



(c) 1288°C (2350°F)

Figure 7. Light photomicrographs of Task I (powder metallurgy) Series I IN-100 alloy after HIP at various temperatures for 4 hours and 103.4 MPa (15 ksi). Magnification ~ 100X

(a) Tensile Test Results

Tensile tests were conducted at 927°C (1700°F) on three specimens from each of the Series I powder metallurgy alloys. The results of these tests are listed in Table II. These results indicated that the TRW-NASA VIA alloy exhibited the highest ultimate tensile and 0.2% offset yield strengths of the four powder alloys. Two of the tensile test specimens exceeded the 517 MPa (75 ksi) program yield strength goal and on the average this alloy exhibited a yield strength level of 516.5 MPa (74.9 ksi). The ductility levels obtained for this material were comparable to those reported in the literature for cast TRW-NASA VIA (29). Following the TRW-NASA VIA alloy, TAZ-8A exhibited the next best combination of tensile strength properties. Both the ultimate tensile strengths and the yield strength values, however, were below those for VIA. The yield strength values, for example, ranged from approximatley 413.7-455.1 MPa (60-66 ksi) and were considerably lower than the average value for the TRW-NASA VIA alloy. The single specimen of IN-100, HIP'ed at 1288°C (2350°F) and given the 24 hour age at 899°C (1650°F) exhibited ultimate tensile and yield strength values which were comparable to the TAZ-8A, but the material HIP'ed at the lower temperature and given the standard solution and age type heat treatment was inferior both in terms of ultimate tensile and yield strength properties. The IN-792 powder also exhibited poor tensile properties, with the yield strengths ranging from approximately 358.5-413.7 MPa (52-60 ksi), depending upon the heat treatment. Application of a 899°C (1650°F)/10 hour carbide stabilization after HIP resulted in the lower yield strength values, whereas a 1121°C (2050°F)/2 hour + 871°C (1600°F)/24 hour partial solution treatment and age resulted in the higher yield strength values.

On the basis of the tensile test results the TRW-NASA VIA exhibited the greatest potential to serve as an alloy base for the alloy development efforts for the powder metallurgy alloys. HIP consolidation at 1316°C (2400°F)/103.4 MPa (15 ksi)/4 hours followed by a 899°C (1650°F)/24 hour age resulted in ultimate tensile strengths approximately 69 MPa (20 ksi) and yield strengths approximatley 35 MPa (10 ksi) higher than the second best of the Series I alloys.

(b) Creep-Rupture Test Results

Creep-rupture tests were conducted at 954°C (1750°F)/206.8 MPa (30 ksi) on five specimens from each of the Series I alloys. The results of these tests are listed in Table III. Similar to the tensile results, the TRW-NASA VIA alloy also exhibited the best overall creep-rupture properties of the Series I powder metallurgy alloys. The TRW-NASA VIA specimens had an average rupture life of 35 hours, approximately double that of the IN-792 and IN-100 (HIP'ed at 1288°C (2350°F)) alloys, the next best of the powder metallurgy alloys. In general, however, the lives to reach 0.1% creep were poor for all of the alloys. The lives for TRW-NASA VIA ranged from 0.22-1.63 hours, while the lives for IN-100 HIP'ed at 1288°C (2350°F) ranged from 0.45-0.62 hours. The TAZ-8A and IN-792 alloys were particularly poor in that neither of these alloys reached 0.2 hours of life to 0.1% creep. For all of the alloys the times to reach 0.1% creep were below the anticipated program goal of 30 hours. These data did, however, indicate certain trends with respect to grain size and subsequent heat treatments after the HIP operations. In those instances in which grain size was relatively

Table II

Task I (Powder Metallurgy Superalloys) Series I 927°C (1700°F) Tensile Test Results

Alloy	Processing Condition	Ultimate Tensile Strength MPa	Ultimate Tensile Strength Ksi	Yield Strength MPa	Yield Strength Ksi	% Elongation	% Reduction Area
TRW-NASA VIA	HIP Condition 1 Plus Heat Treat Condition 1	670.9	97.3	552.3	80.1	3.8	4.9
		673.0	97.6	517.8	75.1	5.7	3.8
		676.4	98.1	749.9	69.6	4.3	5.0
TAZ-8A	HIP Condition 2 Plus Heat Treat Condition 1	532.3	77.2	456.5	66.2	1.9	1.2
		536.5	77.8	458.6	66.5	2.0	2.5
		486.8	70.6	418.6	60.7	1.2	1.2
IN-792	HIP Condition 1 Plus Heat Treat Condition 1	528.2	76.6	370.3	53.7	10.3	13.8
		506.8	73.5	359.9	52.2	9.4	11.5
	HIP Condition 1 Plus Heat Treat Condition 2	415.1	60.2	536.5	77.8	13.4	16.2
IN-100	HIP Condition 3 Plus Heat Treat Condition 3	446.9	64.8	346.8	50.3	2.0	2.0
		448.2	65.0	355.8	51.6	1.9	2.0
	HIP Contion 2 Plus Heat Treat Condition 4	517.1	75.0	448.2	65.0	3.3	2.8

Air Force Program Goal - 517 MPa (75 Ksi) 0.2% offset yield strength at 927°C (1700°F) in a section size of 2.8 cm (7 inches)

HIP Conditions

1. 1316°C (2400°F)/103 MPa (15 Ksi)/4 Hrs
2. 1288°C (2350°F)/103 MPa (15 Ksi)/4 Hrs
3. 1010°C (1850°F)/103 MPa (15 Ksi)/4 Hrs

Heat Treat Conditions

1. 899°C (1650°F)/24 Hrs/Air Cool
2. 1121°C (2050°F)/2 Hrs/Air Cool + 871°C (1600°F)/24 Hrs. Air Cool
3. 1204°C (2200°F)/4 Hrs/Air Cool + 1080°C (1975°F)/4 Hrs/Air Cool + 871°C (1600°F)/16 Hrs/Air Cool + 760°C (1400°F)/24 Hrs/Air Cool

Table III

Task I (Powder Metallurgy) Series I 954°C (1750°F)/206.8 MPa (30 Ksi) Creep Rupture Test Results

Alloy	Processing Condition	Hours to Failure	Hours to 0.1% Creep	% Elongation	% Reduction Area
TRW-NASA VIA	HIP Condition 1 Plus	41.6	1.63	2.6	1.2
	Heat Treat Condition 1	32.9	0.66	1.6	1.6
		47.3	0.28	3.1	1.8
		38.7	0.95	3.8	0.6
		14.3	0.22	2.8	3.1
TAZ-8A	HIP Condition 2 Plus	2.0	0.01	2.0	1.8
	Heat Treat Condition 1	0.9	0.01	2.5	1.2
		1.0	0.01	2.4	2.4
		2.0	0.02	2.1	1.2
		1.2	0.01	2.2	1.9
IN-792	HIP Condition 1 Plus	13.5	0.03	5.5	8.6
	Heat Treat Condition 1	14.6	0.03	8.6	6.8
		22.7	0.03	9.6	7.9
	HIP Condition 1 Plus	15.2	0.03	8.8	7.3
	Heat Treat Condition 2	13.3	0.20	7.4	8.9
IN-100	HIP Condition 3 Plus	0.5	0.01	1.3	2.0
	Heat Treat Condition 3	0.5	0.02	2.1	2.4
		1.0	0.04	2.0	1.9
	HIP Condition 2 Plus	19.5	0.62	3.5	3.1
	Heat Treat Condition 4	10.8	0.45	4.0	2.9
AFML Program Goal			30.0		

HIP Conditions

1. 1316°C (2400°F)/103 MPa (15 Ksi)/4 Hrs
2. 1288°C (2350°F)/103 MPa (15 Ksi)/4 Hrs
3. 1010°C (1850°F)/103 MPa (15 Ksi)/4 Hrs

Heat Treat Conditions

1. 899°C (1650°F)/24 Hours/Air Cool
2. 1121°C (2050°F)/2 Hours/Air Cool +
   871°C (1600°F)/24 Hours/Air Cool
3. 1204°C (2200°F)/4 Hrs/Air Cool +
   1080°C (1975°F)/4 Hrs/Air Cool +
   871°C (1600°F)/16 Hrs/Air Cool +
   760°C (1400°F)/24 Hrs/Air Cool

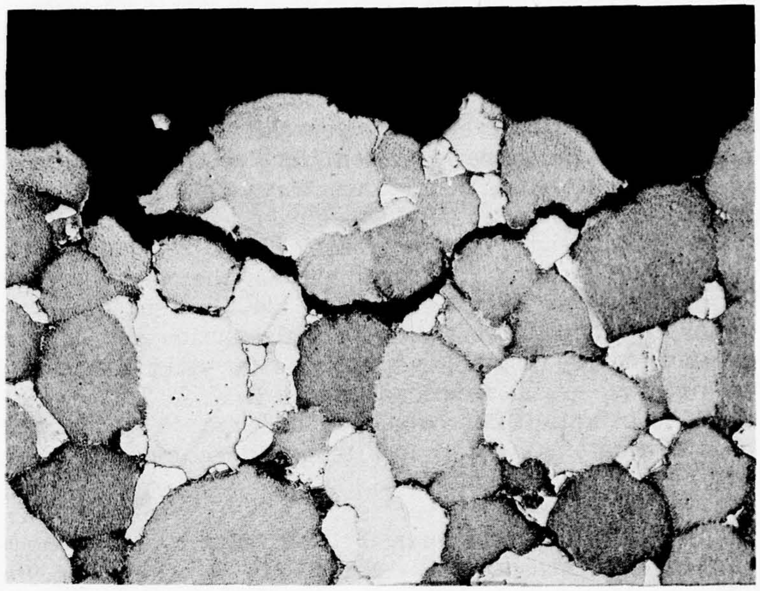
small (e.g., the TAZ-8A alloy and the IN-100 alloy HIP'ed at 1010°C (1850°F)) the rupture lives and the times to 0.1% creep were generally lower in comparison to the larger grained material. This was particularly evident for the IN-100 superalloy in that rupture lives were only approximately one hour. This positive effect of large grain size upon the high temperature creep-rupture properties has been well documented in nickel-base superalloy powders (41). Some insight into the effect of heat treatment variations was gained by the results of the IN-792 alloy. Test specimens for the mechanical property evaluations were obtained from material HIP'ed at 1316°C (2400°F) and given either a low temperature carbide stabilization treatment or a partial solution and age treatment. Whereas the partial solution and age resulted in slightly better yield strengths, Table II, there was little difference in the creep-rupture properties for IN-792. This suggested that there was little likelihood of substantial improvements in time to reach 0.1% creep through heat treatment modifications to the low temperature carbide stabilization applied to the HIP'ed material.

In summary, the creep-rupture test results substantiated the tensile results in identifying TRW-NASA VIA as exhibiting the best combination of mechanical properties consistent with the program target goals. The creep rupture lives for the alloy doubled those of the other Series I powder metallurgy alloys. Although the times to reach 0.1% creep were also superior to the other alloys, the values exhibited by TRW-NASA VIA were far short of the 30 hour program goal. It was clear that a major improvement in time to reach 0.1% creep was required for the consideration of the powder metallurgy TRW-NASA VIA for isothermal forging die applications

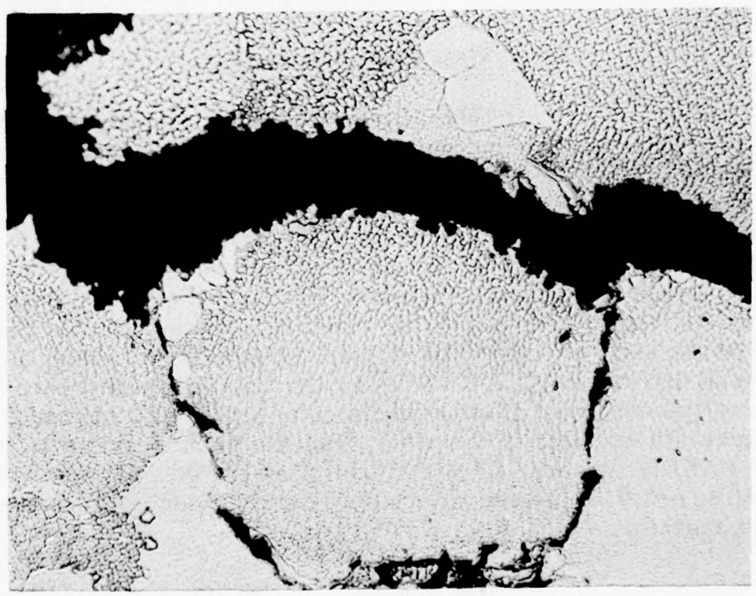
(c) Metallographic Analyses

Metallographic analysis including light metallography and scanning electron microscopy was conducted upon failed test specimens to aid in the interpretation of the mechanical property results. In general, the results indicated that an intergranular failure mode was typical for all the alloys for both the tensile and the creep-rupture tests. This failure mode is commonly observed for nickel-base superalloys tested above 927°C (1700°F) (42). Another feature common to the fracture surfaces was the general absence of porosity or voids. Although the failure mode was similar for all the alloys, the individual fracture paths exhibited characteristics particular to the various alloys. For the large grained TRW-NASA VIA and IN-792 alloys and the large grained IN-100 material HIP'ed at 1288°C (2350°F) the fracture paths were usually associated with the flowery type eutectic gamma-prime colonies located primarily at the grain boundary triple points. An example of a typical fracture path of this type is shown in Figure 8 for the TRW-NASA VIA alloy. As shown in the as-HIP'ed microstructures for these alloys in Figures 4, 6, and 7, a greater amount of this microconstituent was present in the TRW-NASA VIA material.

The fracture paths for the TAZ-8A alloy and the IN-100 material HIP consolidated at 1010°C (1850°F) were somewhat different than the other Series I powder metallurgy alloys. As shown in Figure 5b for the TAZ-8A alloy HIP'ed at 1288°C (2350°F), there was little evidence of the eutectic gamma-prime colonies common to the other alloys. For this particular alloy, however, the fracture paths were usually associated with isolated instances of incipient melting located principally at grain boundary triple points. As also shown in Figure 5, the incidence of this incipient melting



(a) 100X



(b) 500X

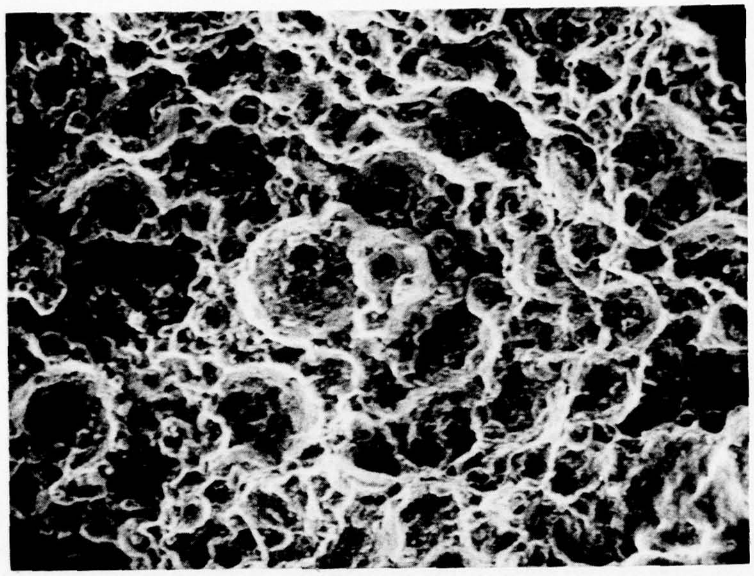
Figure 8. Light photomicrographs of Task I (powder metallurgy) Series I *TRW-NASA VIA alloy showing* intergranular failure mode typical of creep rupture test specimens.

was much less at the 1288°C (2350°F) HIP temperature than at the 1316°C (2400°F) HIP temperature. The IN-100 powder HIP consolidated at 1010°C (1850°F) was the only material to show evidence of fracture along the surfaces of the prior powder particles. The scanning electron fractographs of the IN-100 creep-rupture specimen shown in Figure 9 suggest that the areas of de-bonding may have been the result of employing too low a HIP consolidation temperature. These areas of debonding were not evident in the as-HIP'ed microstructure shown in Figure 7a for IN-100. The poor mechanical property results for this material were not surprising in view of the appearance of the fracture surfaces.

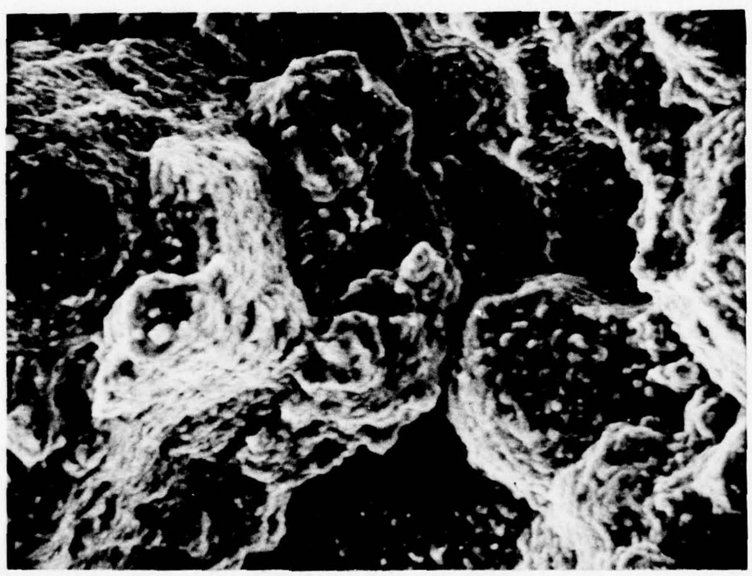
b. Series II Alloy Studies

(1) Alloy Formulation

For the Task I powder metallurgy superalloys, the three aim compositions presented in Table IV were selected for evaluation in Series II. The desired chemistries are presented in terms of weight percent. These alloys were all modifications of TRW-NASA VIA, chosen as the base alloy for Series II because it exhibited the best overall combination of tensile and creep-rupture properties in the Series I mechanical property screening studies. Because of the predominantly intergranular failure mode of the Series I TRW-NASA VIA mechanical property test specimens, Figure 8, these alloy variations were designed to improve the creep-rupture capabilities of the base alloy primarily through grain boundary strenghtening. Compared to the base alloy composition shown in Table I, Modification I was aimed at increased hafnium (1.6% versus 0.43%), rhenium added at the 0.22% level and reduced carbon, boron and zirconium contents. The hafnium content was increased in order to obtain the improvements in elevated temperature rupture life, ductility and tensile strength reported for cast TRW-NASA VIA for similar hafnium levels (43). These improvements were attributed to the changes in the cast microstructure resulting from the hafnium additions and included the formation of blocky gamma-prime grain boundary precipitates and a change in carbide morphology from a script-like shape to a blocky type shape. Hafnium additions to nickel-base superalloy powder alloys have also resulted in improvements in elevated temperature mechanical properties, primarily by altering the gamma-prime in these alloys from a continuous film-like grain boundary morphology to a more discrete particle type precipitate (44). The carbon, boron and zirconium aim levels were reduced in Modification I compared to the Series I TRW-NASA VIA because of the reported improvements in creep-rupture life with these modifications in other nickel-base superalloys (45,46). Rhenium was added to the alloy because it has resulted in some grain boundary strengthening (30) and has recently been used as a potent alloy strengthener for advanced superalloy turbine blade compositions (47). Modification II had the hafnium aim level increased to 2.5% to explore the benefits of higher hafnium content and had tantalum reduced to 6% from 9% in order to balance the increase in gamma-prime content anticipated by the hafnium increase. Rhenium was also added to this alloy at the 0.22% aim level and the carbon, boron and zirconium contents were lowered to the Modification I aim levels. Modification III was a hafnium and rhenium free variation of TRW-NASA VIA reported to have a substantial creep life advantage over the conventional TRW-NASA VIA composition (48). This creep life increase was realized through the tailoring of the gamma/gamma-prime lattice mismatch.



(a) 200X



(b) 1000X

Figure 9. Scanning electron fractographs of creep rupture specimen of Task I (powder metallurgy) Series I IN-100 alloy HIP'ed at 1010°C (1850°F). Note areas of de-bonding on the fracture surface.

Table IV

Task I (Powder Metallurgy Superalloys) Series II Alloy Compositions[(1)]

Alloy		C	Cr	Mo	Ti	Al	Co	W	Zr	B	Ta	Cb	Hf	Re	O_2	N_2
TRW-NASA VIA	Aim	0.04	5.6	2.0	1.0	5.4	7.75	6.0	0.01	0.006	9.0	0.5	1.6	0.22		
Modification I	Actual	0.072	5.83	1.92	.89	5.16	7.15	5.61	0.03	0.01	7.90	0.43	1.5	0.24	245	45
TRW-NASA VIA	Aim	0.04	5.6	2.0	1.0	5.4	7.75	6.0	0.01	0.006	6.0	0.5	2.5	0.22		
Modification II	Actual	0.07	5.8	1.95	0.91	5.4	7.50	5.93	0.02	0.01	6.9	0.45	2.29	0.23	110	41
TRW-NASA VIA	Aim	0.10	6.0	1.0	2.0	4.5	10.0	8.5		0.01	6.0	2.0			198	29
Modification III	Actual	0.16	5.96	1.06	2.0	4.58	9.54	8.7		0.009	5.3	2.35				

(1) In weight percent, except for oxygen and nitrogen, which are reported in parts per million.

The actual compositions in weight percent of the material processed during the HIP parametric study are also listed in Table IV. Specimens of this material were used for the mechanical property screening evaluations. Comparison of the aim compositions with the actual chemistries indicated that, with the exception of carbon content, the desired compositions were achieved. For Modifications I and II the carbon contents were 0.03% higher than desired, while for Modification III the carbon was 0.06% higher than the desired level. Particular attention was directed during the HIP parametric study to problems concerning fine grain size in the HIP compacts as the result of the higher carbon contents. One other observation was made concerning the chemical analysis of the Series II alloys and involved the oxygen results. The analyses indicated that the oxygen levels for Modifications I and III were higher than that for the Modification II alloy and in general were also higher than the Series I alloys, Table I. The oxygen content for Modification III approached 200 ppm while that for Modification I was above 200 ppm. During analysis of the fractured mechanical property test specimens attention was paid to the possible detrimental effects of these high oxygen contents in terms of embrittling oxide networks.

(2) HIP Parametric Study

The HIP parametric study was conducted on the three TRW-NASA VIA Series II modifications in order to determine the processing conditions required to achieve complete densification in the consolidated powders and the desired ASTM 2-3 grain size. The selection of an initial 1316°C (2400°F)/103.4 MPa (15 ksi) HIP condition was based on the satisfactory results obtained with the Series I TRW-NASA VIA alloy. The grain size data resulting from this processing condition are given below for the three alloys:

TRW-NASA VIA Modification I	ASTM 1-3
TRW-NASA VIA Modification II	ASTM 2-4
TRW-NASA VIA Modification III	ASTM 2-2

These grain size results compared quite favorably with the ASTM 2-4 values exhibited by the Series I TRW-NASA VIA alloy, Figure 4c, consolidated at the same temperature. It was apparent that the higher carbon contents of the Series II alloys in relation to the Series I TRW-NASA VIA (0.034%) did not prevent the attainment of large grain sizes in the HIP'ed material. In order to explore the possibility of achieving still larger grain sizes by employing higher HIP temperatures, material HIP'ed at 1316°C (2400°F) was heat treated at 1329°C (2425°F) and 1343°C (2450°F) for four hours in an argon filled tube furnace. These thermal exposures resulted in such large amounts of incipient melting at grain boundary triple points that the 1316°C (2400°F) HIP consolidation temperature was selected for all three of the Series II alloys. These heat treatment results also indicated that the gamma-prime precipitates in the as-HIP'ed material could not be completely solutioned without the incidence of appreciable amounts of incipient melting throughout the microstructure. As a result, the 899°C (1650°F)/24 hour carbide stabilization used for the Series I alloy was also employed for the three Series II modifications.

Examples of the microstructures of the HIP'ed and aged Modification I and II alloys are shown in Figure 10. Comparison of these structures with those shown in Figure 4c for as-HIP'ed material and Figure 8 for HIP'ed and aged creep tested material indicates the similarity of grain sizes. Note the isolated instances of incipient melting in the Modification I alloy. The microstructure of the Modification III alloy was similar to this except for slightly larger amounts of incipient melting throughout the microstructure. The structures of the Modification I and III alloys were similar to the Series I alloy in terms of the relative amounts of the flowery-type of eutectic gamma-prime precipitates. Modification II alloy exhibited appreciably less of this microconstituent throughout its microstructure. The high magnification photomicrographs of Figure 10c and d indicate that the grain boundaries were characterized by the presence of discrete particle precipitates of both gamma-prime and carbide phases.

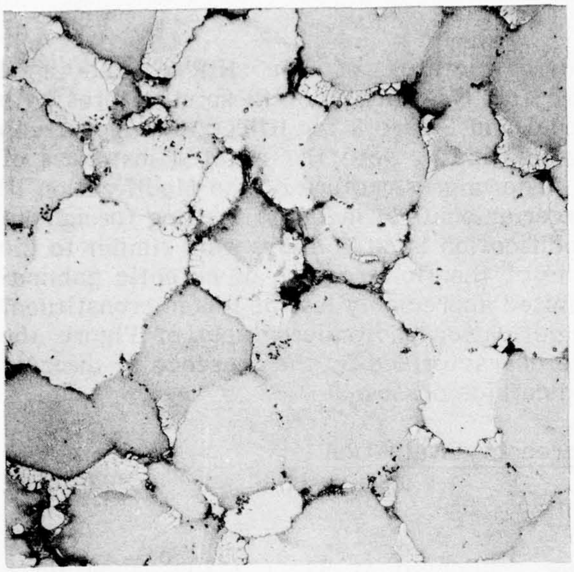
(3) Mechanical Property Screening Evaluation

(a) Tensile Test Results

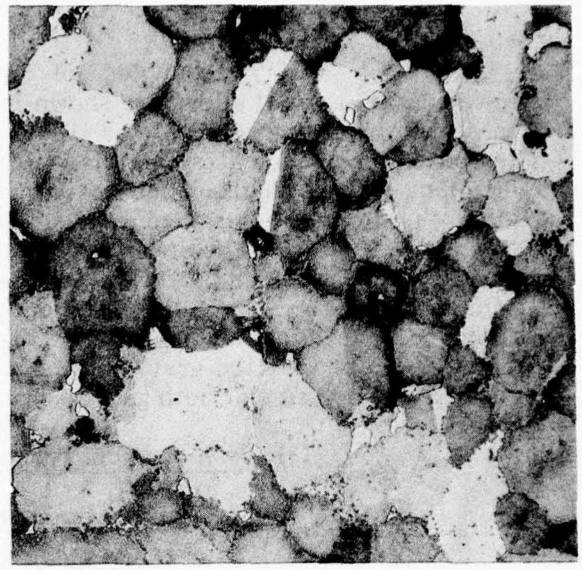
Tensile tests were conducted at 927°C (1700°F) on three specimens from each of the Series II powder metallurgy alloys and the results of these tests are listed in Table V. These tensile results indicated that Modifications I and II consistently achieved the yield strength program goal of 517 MPa (75 ksi). On the average, Modification I exhibited a 557.2 MPa (80.8 ksi) yield strength, while Modification II exhibited a 574.4 MPa (83.3 ksi) yield strength. While Modification III test specimens exhibited yield strengths approximating the program goal, they were generally inferior to the other two alloys. Overall, Modification II offered the best combination of 927°C (1700°F) tensile properties with the highest ultimate tensile strength, yield strength and ductilities of the three Series II alloy. Comparison to the Series I TRW-NASA VIA composition indicated that the increased hafnium, reduced boron and zirconium and the addition of rhenium to the Modification II alloy resulted in approximatley the same ultimate tensile strength, an improvement in yield strength but at a loss in the ductility. This loss in ductility (by approximately 40% in comparison to the Series I TRW-NASA VIA) was difficult to rationalize in terms of chemistry because of the reported ductility improvements obtained with these particular chemistry modifications. A plot of the yield strength for the Series II powder metallurgy alloys compared to the Series I TRW-NASA VIA alloy is shown in Figure 11. All of the yield strength data values were included in this plot for each of the alloys. Both Modifications I and II, involving hafnium increases, rhenium additions and reductions in boron and zirconium, exhibited an improvement in yield strength capability compared to the Series I alloy. On the average, Modification III was comparable in yield strength to the original TRW-NASA VIA powder metallurgy composition.

(b) Creep-Rupture Test Results

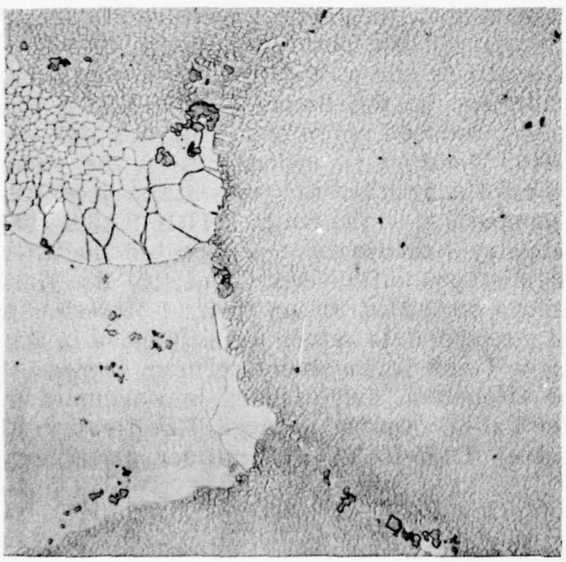
Creep-rupture tests were conducted at 954°C (1750°F)/206.8 MPa (30 ksi) on five specimens from each of the Series II alloys and the results of these tests are listed in Table VI. The results were similar to the tensile tests in that Modification II also offered the best overall creep-rupture properties of the three Series II alloys. This alloy exhibited an average creep-rupture life of 19.8 hours and times to reach 0.1% creep ranging from 0.37-9.5 hours. Modification I, the second best of the Series II



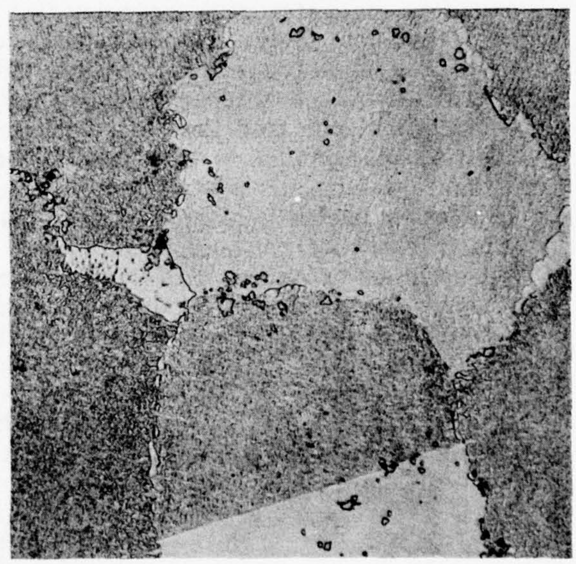
(a) Modification I, 100X



(b) Modification II, 100X



(c) Modification I, 500X



(d) Modification II, 500X

Figure 10. Light photomicrographs of Task I (powder metallurgy) Series II TRW-NASA VIA modifications I and II after HIP at 1316°C (2400°F)/103.4 MPa (15 ksi)/4 hours plus 24 hours age at 899°C (1650°C).

Table V

Task I (Powder Metallurgy Superalloys) Series II 927°C (1700°F) Tensile Test Results[1]

Alloy	Ultimate Tensile Strength MPa	Ultimate Tensile Strength Ksi	0.2% Offset Yield Strength MPa	0.2% Offset Yield Strength Ksi	% Elongation	% Reduction Area
TRW-NASA VIA	587.5	85.2	542.0	78.6	1.1	1.2
Modification I	608.8	88.3	560.6	81.3	1.4	1.8
	637.1	92.4	568.2	82.4	1.5	0.6
TRW-NASA VIA	650.2	94.3	564.8	81.9	3.0	3.8
Modification II	672.3	97.5	580.6	84.2	2.8	1.8
	668.2	96.9	578.6	83.9	2.9	1.8
TRW-NASA VIA	573.0	83.1	502.7	72.9	1.1	1.2
Modification III	603.3	87.5	524.0	76.0	1.5	1.8
	596.4	86.5	498.5	72.3	1.7	1.8

Air Force Program Goal - 517 MPa (75 Ksi) 0.2% offset yield strength at 927°C (1700°F) in a section size of 2.8 cm (7 inches)

(1) Processing conditions for all specimens included HIP at 1316°C (2400°F)/103 MPa (15 Ksi)/4 Hrs plus 899°C (1650°F)/24 hrs/air cool heat treatment.

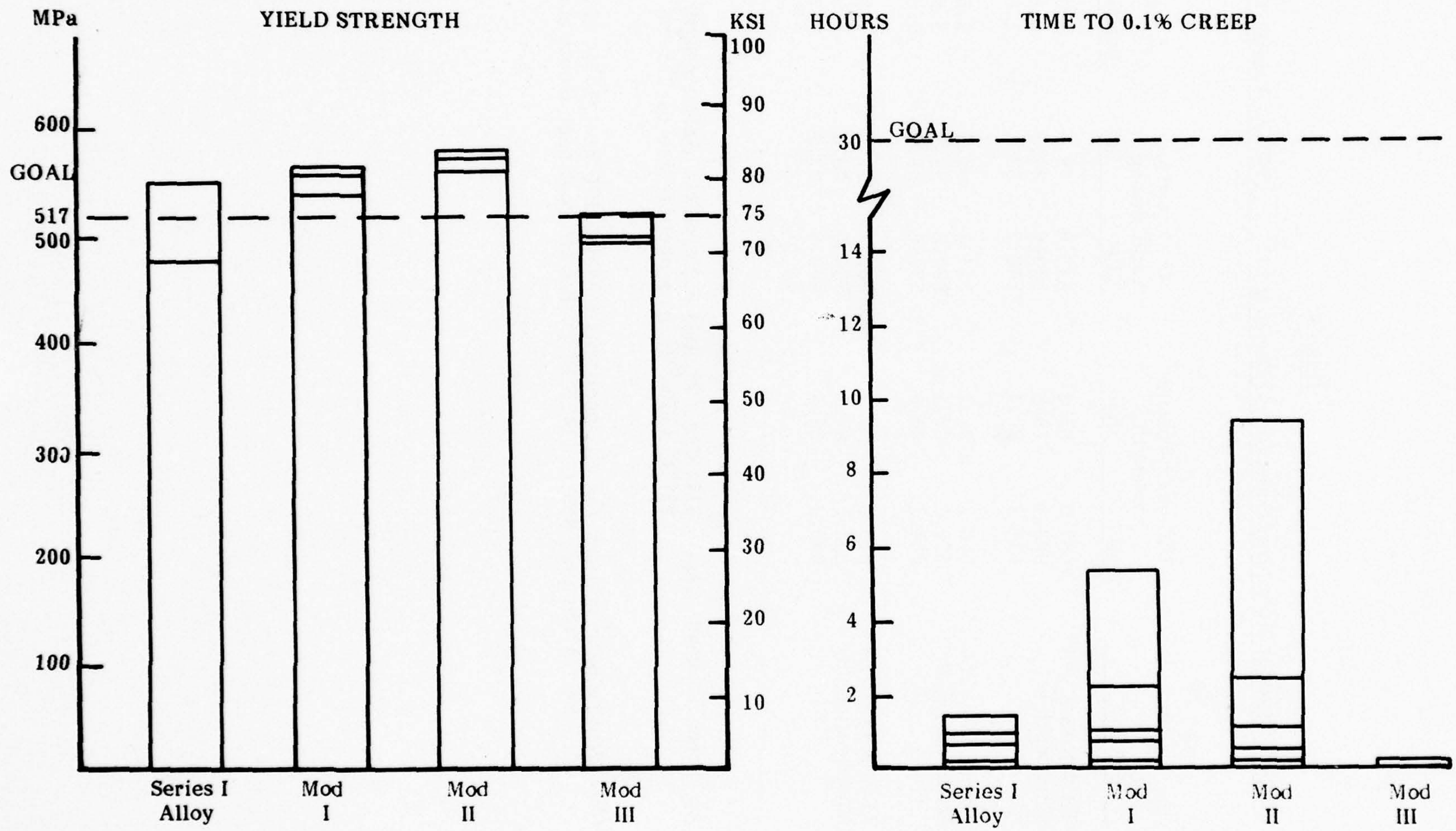


Figure 11. Yield strength and time to 0.1% creep for Task I (powder metallurgy) Series II Alloys compared to Series I TRW-NASA VIA alloy.

Table VI

Task I (Powder Metallurgy Superalloys) Series II $954^{\circ}C$ ($1750^{\circ}F$)/206.8MPa (30 Ksi)

Creep Rupture Test Results[1]

Alloy	Hours to Failure	Hours to 0.1% Creep	% Elongation	% Reduction Area
TRW-NASA VIA	17.0	5.43	1.3	0.3
Modification I	16.1	2.25	1.7	0.8
	21.5	1.07	2.0	1.6
	18.6	0.75	1.7	1.2
	16.2	0.12	1.9	1.2
TRW-NASA VIA	27.7	9.50	1.7	1.2
Modification II	26.5	2.43	2.1	0.4
	6.6	1.17	0.7	0.0
	19.9	0.56	1.2	0.8
	18.1	0.37	1.5	0.8
TRW-NASA VIA	16.8	0.23	3.3	2.7
Modification III	15.6	0.10	2.8	3.1
	13.7	0.06	2.9	3.1
	13.3	0.04	4.0	3.1
	(2)			
AFML Program Goal		30.0		

(1) Processing conditions for all specimens included HIP at $1316^{\circ}C$ ($2400^{\circ}F$)/103 MPa (15 Ksi)/4 Hrs plus $899^{\circ}C$ ($1650^{\circ}F$)/24 Hrs/air cool heat treatment.

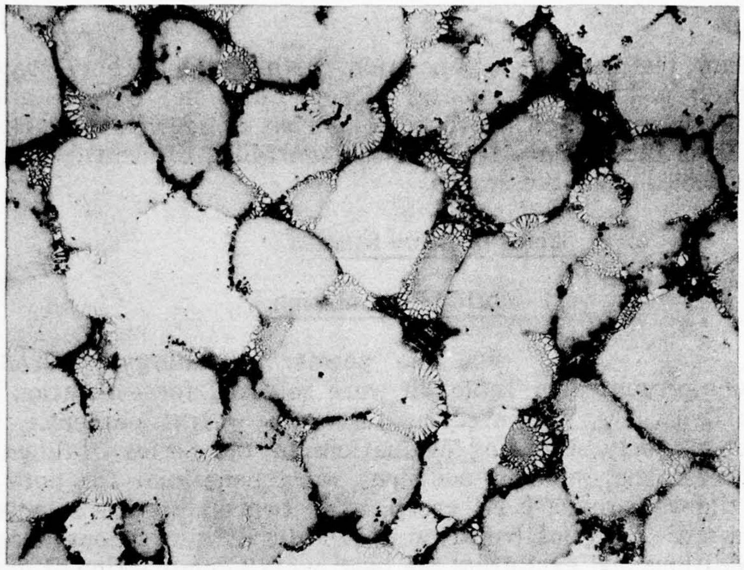
(2) Specimen failed on loading.

alloys, exhibited an average creep-rupture life of 17.9 hours and times to reach 0.1% ranging from 0.12-5.43 hours. The Modification III alloy was characterized by an average creep-rupture life of 14.9 hours and by times to reach 0.1% creep ranging from 0.04-0.23 hours. Comparison to the Series I TRW-NASA VIA composition indicated that the increased hafnium, reduced boron and zirconium and the addition of rhenium to the Modification I and II alloys resulted in improvements in the times to reach 0.1% creep for the Series II alloys. These improvements are shown in Figure 11, which includes a plot of all the test values of time to reach 0.1% creep for the Series II alloys compared to the Series I base composition. The relatively poor response of the Modification III alloy is also evident in this figure. It was significant to note that while the hafnium, boron, zirconium and rhenium chemistry modifications improved the times to reach 0.1% creep compared to the TRW-NASA VIA base composition, the creep-rupture failure times and the rupture ductilities were inferior for the Series II modified alloys. This suggested that while the chemistry modifications may have acted to influence creep deformation mechanisms such as grain boundary sliding, once cracks had been initited, their propagation to eventual failure was rapid in comparison to the base composition alloy.

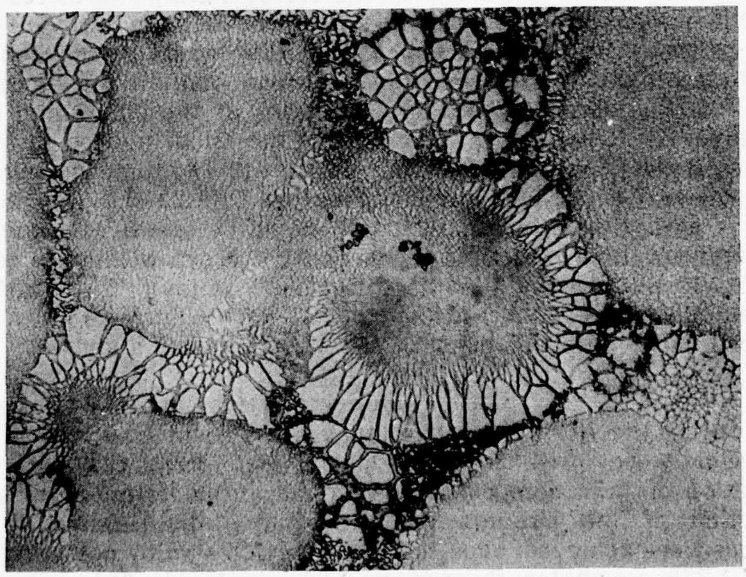
(c) Metallographic Analysis

Metallographic analysis conducted upon failed test bar specimens indicated that, in general, an intergranular failure mode was common for all three alloys for both the tensile and the creep-rupture tests. The fracture paths for the Series II alloys were similar to those of the Series I TRW-NASA VIA composition in that they were usually associated with the flowery-type eutectic gamma-prime colonies located primarily at grain boundary triple points. As shown in Figure 10 for Modifications I and II and in Figure 12 for the Modification III alloy, the relative amount of this microconstituent was far less in the Modification II alloy than in either of the other two compositions. This may, in part, have been responsible for the superior creep-rupture properties of the Modification II alloy. The morphology of this phase appeared particularly undesirable in the Modification III alloy. As shown in the high magnification photomicrograph of Figure 12b, the microstructure of Modification III alloy was characterized by an almost continuous eutectic gamma-prime phase surrounding grains, which also contained considerable evidence of incipient melting. This morphology was probably responsible for the poor creep-rupture properties exhibited by the alloy.

It was difficult to rationalize the mechanical property differences between the Modification II alloy (the best of the Series II alloys) and the Series I TRW-NASA VIA composition strictly on the basis of microstructural differences. Modification II offered comparable ultimate tensile strength, improved yield strength and time to reach 0.1% creep, but at a loss in ductility and creep-rupture failure time compared to the Series I alloy. A comparison of the microstructures of the Modification II material, shown in Figure 10, with that of the Series I composition, shown in Figure 8, indicate that the Modification II alloy contained appreciably less eutectic gamma-prime colonies than the Series I alloy. Since fracture appeared to be intimately associated with this microconstituent, this microstructural change overall should have been beneficial to the mechanical properties. That it was not, suggested that some other factor was also playing a prominent role in the mechanical property behavior. One such possibility may have been the higher oxygen content of the Modification II alloy compared to the Series I material. In general, it was observed that all of the Series II alloys were higher in oxygen



(a) 100X



(b) 500X

Figure 12. Light photomicrographs of Task I (powder metallurgy) Series II TRW-NASA VIA Modification III creep-rupture test specimen.

content than the Series I composition. While the higher oxygen contents remained suspect, their possible detrimental effects could not be substantiated experimentally, however, because electron microprobe analysis of secondary cracks within the specimens and away from the primary fracture surface failed to identify substantial concentrations of oxide particles along the cracks.

c. Series III Alloy Studies

(1) Alloy Formulation

For the powder metallurgy superalloys, the two aim compositions presented in Table VII were selected for evaluation in Series III. These aim chemistries are presented in terms of weight percent. The results of the mechanical property screening evaluations of the Series II alloys indicated that high hafnium levels and rhenium additions were beneficial for both yield strength and improved times to reach 0.1% creep. The two alloys thus selected for evaluation in Series III were variations based on the Series II alloys containing high hafnium and rhenium additions and exhibiting the best overall combination of mechanical properties. The Modification IV alloy was aimed at a very high hafnium level (2.5%) compared to the highest hafnium containing alloy of the Series II compositions (Modification II at 2.29%). This high hafnium level was selected to explore the possible benefits of increased amounts of this element. Modification IV also featured a reduced carbon level (0.02% compared to the 0.04% of the Series II alloys) to increase the effectiveness of the hafnium present by reducing the amount of hafnium carbide formed in preference to gamma-prime. The tantalum aim in this alloy was also reduced from 9% to 6% in order to balance the increase in gamma-prime content anticipated by the hafnium increase. Rhenium was also added to this alloy at the 0.22% aim level and the boron and zirconium contents were lowered to the Modification I and II levels. The Modification V alloy featured a hafnium content (2.0%) somewhat intermediate between that of Modification I (1.5%) and Modification II (2.29%). The objective here was to determine if an optimum hafnium content could be identified which offered enhanced yield strength and improved time to reach 0.1% creep within the range 0.51% (Series I TRW-NASA VIA alloy) and a 2.5% (Modification IV alloy). The Modification V alloy also featured the low boron and zirconium contents of the Series II alloys as well as the 9.0% tantalum and 0.04% carbon contents of the Modification I alloy.

The actual compositions in weight percent of the material processed during the HIP parametric study are also listed in Table VII. Specimens of this material were used for the mechanical property characterization studies. Comparison of the aim compositions with the actual chemistries indicated that, with the exception of the hafnium content of the Modification V alloy, the desired compositions were achieved. For the Modification V alloy however, the actual hafnium content of 1.77% was below the desired 2.0% aim level. This composition was used for the Series III evaluations, however, because the hafnium composition was intermediate between the 1.5% level of the Modification I alloy and the 2.29% level of the Modification II alloy and as such represented the opportunity to have an evaluation conducted on a series of alloys ranging in hafnium content from 0.51% (Series I alloy) to 2.43% (Modification IV). A further observation concerning the chemical analyses of the

Table VII

Task I (Powder Metallurgy Superalloys) Series III Alloy Compositions(1)

Alloy		C	Cr	Mo	Ti	Al	Co	W	Zr	B	Ta	Cb	Hf	Re	O_2	N_2
TRW-NASA VIA	Aim	0.02	5.6	2.0	1.0	5.4	7.75	6.0	.01	.006	6.0	0.5	2.5	0.22		
Modification IV	Actual	0.024	5.86	2.05	.86	5.51	7.63	6.05	.04	.009	5.74	0.51	2.43	0.21	95	39
TRW-NASA VIA	Aim	0.04	5.6	2.0	1.0	5.4	7.75	6.0	.01	.006	9.0	0.5	2.0	0.22		
Modification V	Actual	0.049	5.51	2.07	.80	5.41	7.44	5.84	.03	.007	7.9	0.48	1.77	0.20	85	28

(1) In weight percent, except for oxygen and nitrogen, which are reported in parts per million.

Series III alloys involved the oxygen levels. For both alloys the oxygen contents were below 100 ppm, and were thus comparable to the Series I TRW-NASA VIA composition, Table I. By comparison, Table IV, the Series II oxygen levels were somewhat higher.

(2) HIP Parametric Study

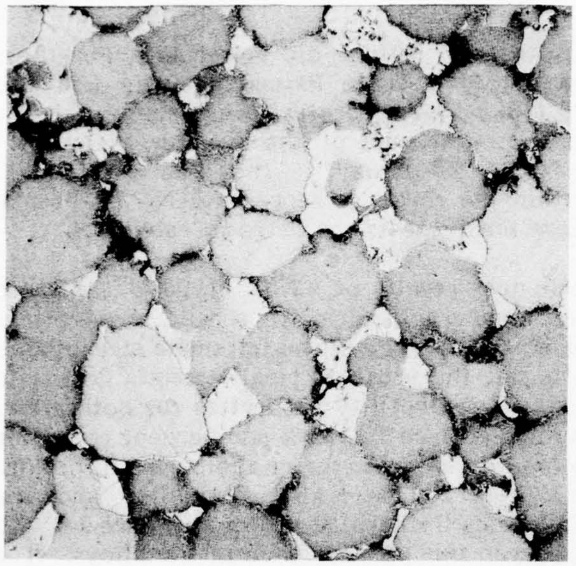
The HIP parametric study was conducted on the two Series III powder metallurgy alloys in order to establish the processing conditions required to achieve complete densification and the ASTM 2-3 grain size necessary for high temperature creep-rupture properties. The selection of an initial 1316°C (2400°F)/103.4 MPa (15 ksi)/4 hour HIP condition was based upon the satisfactory results achieved with the Series I TRW-NASA VIA alloy as well as the Series II compositions. The grain size measurements resulting from those processing conditions indicated ranges of ASTM 2-4 grains, and compared quite favorably with the grain sizes of the Modification II alloy (the best of the Series II alloys) shown in Figure 10a. Complete densification was achieved as the result of this HIP operation. Thermal exposures of this HIP'ed material at 1329°C (2425°F) for four hours in an argon filled tube furnace resulted in some additional grain growth but with substantial amounts of accompanying incipient melting. These thermal exposures also indicated that the gamma-prime precipitates in the as-HIP'ed material could not be completely solutioned without the incidence of appreciable amounts of incipient melting throughout the microstructure. As a result of these considerations the 1316°C (2400°F) HIP consolidation temperature was selected for the Series III alloys as well as the 899°C (1650°F)/24 hour carbide stabilization heat treatment used for the Series I and II alloys.

Examples of the HIP'ed and aged Modification IV and V powder metallurgy alloys are shown in Figure 13. Comparison of these structures with those shown in Figure 4c for as-HIP'ed Series I alloy and Figures 10a and b for the HIP'ed and aged Modification I and II alloys indicates the overall similarity in the microstructures of these alloys. The grain sizes, in general, were similar, ranging from ASTM 2-4 depending upon the particular alloy composition. The high magnification photomicrographs indicate that the grain boundaries were characterized by the presence of discrete particle precipitates of both gamma-prime and carbide phases. The sizes and distributions of the gamma-prime eutectic colonies were also similar, in general, with the exception of Modification II alloy, in which slightly less of this microconstituent was present throughout the structure.

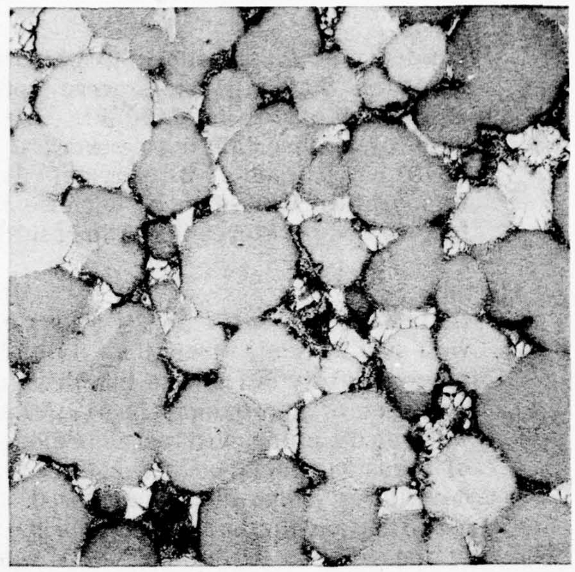
(3) Mechanical Property Characterization

(a) Tensile Test Results

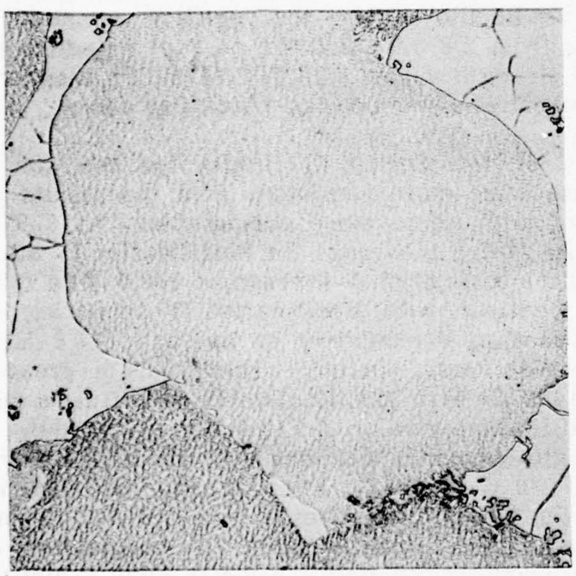
Tensile tests were conducted on five specimens from each of the Series III powder metallurgy alloys. Initially, duplicate specimens were tested at 927°C (1700°F). When comparison of the results with those for the Modification II alloy (the best of the Series II powder metallurgy alloys) indicated little improvement in ultimate tensile strength or 0.2% yield strength, the remaining three specimens were tested at other temperatures within the anticipated range of isothermal forging conditions to provide more information as to the potential capability of the



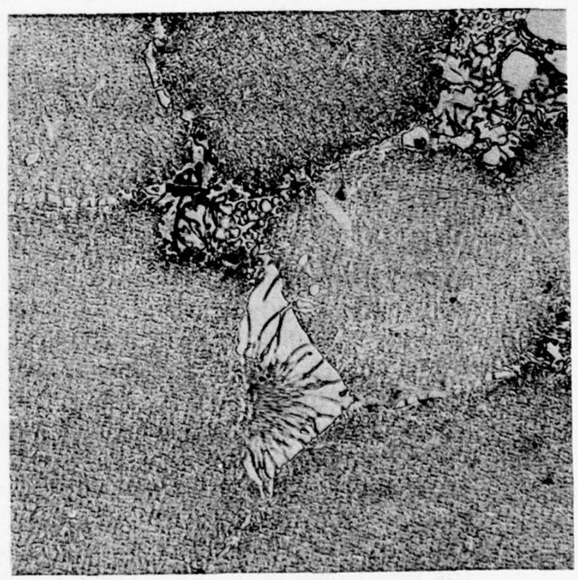
(a) Modification IV, 100X



(b) Modification V, 100X



(c) Modification IV, 500X



(d) Modification V, 500X

Figure 13. Light photomicrographs of Task I (powder metallurgy) Series III TRW-NASA VIA Modifications IV and V after HIP at 1316°C (2400°F)/103.4 MPa (15 ksi) 4 hours plus 24 hours age at 899°C (1650°F).

powder metallurgy alloys to perform as isothermal forging die materials. For these purposes, duplicate tests were conducted at 830°C (1525°F) and a single test was conducted at 760°C (1400°F). The 830°C (1525°F) temperature was selected on the basis of isothermal forging work to be conducted as part of the Air Force Contract F33615-76-C-5386, "Isothermal Forging Beta Titanium." The 760°C (1400°F) temperature was selected because it is within the range of representative isothermal forging temperatures (5). The results of these tensile tests are listed in Table VIII.

The tensile test results at 927°C (1700°F) indicated that Modification IV and V alloys exhibited comparable ultimate tensile strengths as well as 0.2% yield strengths. Both alloys exhibited average ultimate tensile strengths of approximately 661.9 MPa (96.0 ksi) and both met the program yield strength goal with averages of approximately 517 MPa (75 ksi). The ductility properties for both alloys were also similar, with percent elongations ranging from 1.5-1.9% and percent reduction of areas ranging from 2.0-2.4%. A plot of the yield strength for the Series III powder metallurgy superalloys compared to the Series I TRW-NASA VIA alloy and the Modification II composition (the best of the Series II alloys) is shown in Figure 14. All of the yield strength data values were included in this plot for each of the alloys. It is evident from this plot that while both Modification IV and V alloys exhibited comparable average yield strength values and met the program goal of 517 MPa (75 ksi), they were somewhat inferior to the Modification II composition. This alloy exhibited an average yield strength of 574.4 MPa (83.3 ksi), which exceeded the program goal. As shown in Table V for the Series II tensile data, the ultimate tensile strength of the Modification II alloy was comparable to that of the Series III alloys while the ductility was slightly superior, with percent elongation ranging from 2.8-3.0% and the percent reduction of area ranging from 1.8-3.8%. In terms of strength values at 927°C (1700°F), then, the Modification II alloy exhibited the best properties of the powder metallurgy alloys.

Analysis of the Series III results for the 830°C (1525°F) and 760°C (1400°F) tensile data were quite consistent with the results at 927°C (1700°F) in that the properties of both alloys were comparable. At 830°C (1525°F), for example, the ultimate tensile strength average for Modification IV alloy was 883.9 MPa (128.2 ksi) compared to the Modification V average of 868.7 MPa (126 ksi). The yield strengths were almost identical, with Modification IV exhibiting an average 710.9 MPa (103.1 ksi) and Modification V exhibiting an average 717.8 MPa (104.1). The ductility properties were also quite similar. There was a greater difference in yield strengths at 760°C (1400°F) with Modification IV exhibiting a 866 MPa (125.6 ksi) value compared to the Modification V value of 719.9 MPa (104.4 ksi). It must be noted, however, that only a single specimen was tested at 760°C (1400°F), while duplicate tests were conducted at 830°C (1525°F). As a group, the Series III powder metallurgy alloys exhibited inferior tensile properties at 830°C (1525°F) and 760°C (1400°F) compared to the published values for TRW-NASA VIA composition in the cast form (29). As a comparison, for example, the Modification IV alloy exhibited an average yield strength of 710.9 MPa (103.1 ksi) at 830°C (1525°F) compared to the published value of 827.4 MPa (120 ksi) for cast TRW-NASA VIA. This particular yield strength level is approximately 15% below the value of the cast material. In general, all of the tensile strength properties of the Series III powder metallurgy alloys were approximately 15% below the values published for cast TRW-NASA VIA. The published

Table VIII

Task I (Powder Metallurgy Superalloys) Series III Tensile Test Results[1]

Alloy	Test Temperature °C	°F	Ultimate Tensile Strength MPa	Ksi	0.2% Offset Yield Strength MPa	Ksi	% Elongation	% Reduction Area
TRW-NASA VIA	927	1700	671.6	97.4	514.4	74.6	1.4	1.8
Modification IV	927	1700	668.2	96.9	534.4	77.5	1.6	2.1
	830	1525	903.9	131.1	744.0	107.9	9.6	6.2
	830	1525	863.9	125.3	677.8	98.3	8.0	4.9
	760	1400	1038.9	150.7	866.0	125.6	2.5	6.3
TRW-NASA VIA	927	1700	668.8	97.0	515.8	74.8	1.8	2.3
Modification V	927	1700	658.5	95.5	519.3	75.3	2.0	2.4
	830	1525	892.6	129.6	737.1	106.9	10.4	7.7
	830	1525	844.0	122.4	698.5	101.3	6.9	5.3
	760	1400	1028.4	149.2	719.9	104.4	4.6	7.4

Air Force Program Goal: 517 MPa (75 Ksi) 0.2% offset yield strength at 927°F (1700°F) in a section size of 2.8 cm (7 inches).

(1) Processing included HIP consolidation at 1316°C (2400°F)/103 MPa (15 Ksi)/4 hours plus heat treatment of 899°C (1659°F)/24 hours/air cool.

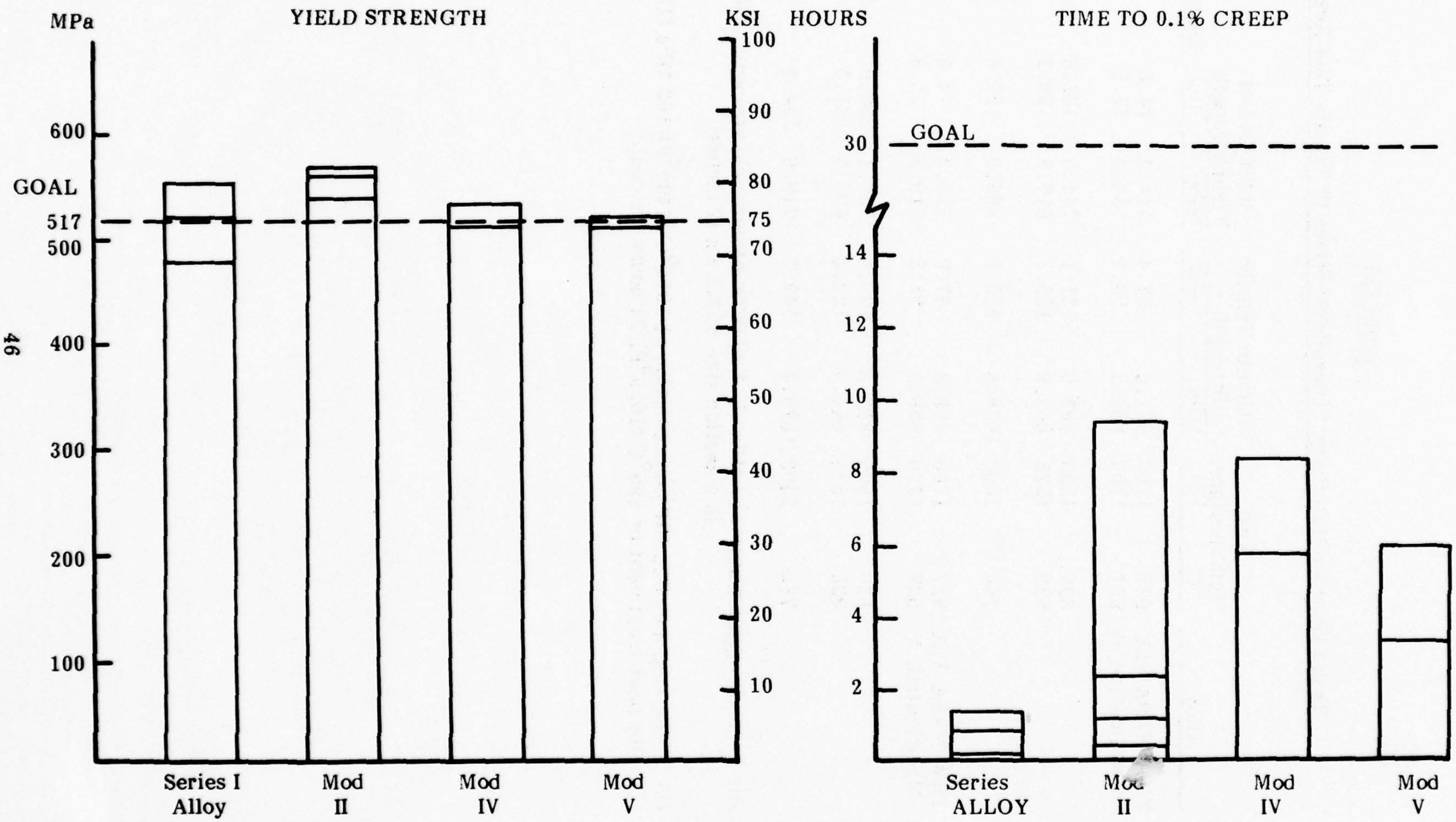


Figure 14. Yield strength and time to 0.1% creep for the Task I (powder metallurgv) Series III alloys compared to Series I TRW-NASA VIA and Series II Modification II alloys.

values were obtained from cast-to-size test specimens and as such a certain reduction is anticipated in thick sections because of section size effects. As will be discussed in the Task II casting superalloy efforts, however, the reduction in tensile properties exhibited by the powder metallurgy alloys was similar to that exhibited by the thick section casting alloy test specimens when compared to the published values for cast to size specimens. The tensile results, in general then, support the contention that the powder metallurgy superalloys exhibit potential for application as isothermal forging die materials.

(b) Creep-Rupture Test Results

Creep-rupture tests were conducted on seven specimens from each of the Series III alloys and the results of these tests are listed in Table IX. Initially, three tests were conducted for each alloy at 954°C (1750°F)/206.8 MPa (30 ksi). When these initial results indicated little improvement in the creep-rupture properties compared to the Modification II alloy (the best of the Series II compositions), the remaining four specimens were tested at temperatures similar to those of the tensile tests to provide more information concerning the potential capability of powder metallurgy superalloys for isothermal forging die applications. These test temperatures included 830°C (1525°F) and 760°C (1400°F).

The creep-rupture results at 954°C (1750°F) indicated that the Modification IV alloy offered only slightly better properties compared to the Modification V alloy. Modification IV averaged rupture lives of 22.0 hours compared to the 17.7 hour average for Modification V and exhibited times to reach 0.1% creep ranging from 2.5-8.4 hours, compared to the 3.4-6.0 hour range for the Modification V alloy. The ductility properties were somewhat inconsistent in that Modification IV exhibited better reduction of area but poorer percent elongation. In general, these creep rupture properties indicated little improvement in comparison to the properties offered by the Modification II alloy (the best of the Series II compositions). The average rupture life for Modification II was 19.8 hours and was comparable to the average values exhibited by the Series III compositions. The Modification II alloy exhibited a somewhat higher maximum value for time to reach 0.1% creep, with times ranging from 0.37-9.5 hours. These creep-rupture results are shown in Figure 14, which includes a plot of all the test values of time to reach 0.1% creep for the Series III alloys compared to the Series I base composition and the Modification II alloy. Only those tests conducted at 954°C (1750°F) were included in this plot. These results indicated that the chemistry modifications employed for the Series II and III compositions did improve the creep rupture time to reach 0.1% creep for the original Series I material. Hafnium increases to approximately 2.29% (Modification II alloy) and rhenium additions up to 0.20% were particularly helpful in this regard. There appeared to be little beneficial effect by adding hafnium at a higher content (the 2.43% for the Modification IV alloy). In spite of the improvements compared to the base TRW-NASA VIA Series I composition, however, the ranges of times to reach 0.1% creep exhibited by the powder metallurgy alloys were considerably short of the program goal of 30 hours. A single specimen of Modification II alloy exhibited a maximum of 9.5 hours to reach 0.1% creep while a specimen of Modification IV alloy exhibited a time of 8.4 hours.

Table IX

Task I (Powder Metallurgy Superalloys) Series III

Creep Rupture Test Results (1)

Alloy	Test Temperature °C	°F	Stress Level MPa	Ksi	Hours to Failure	Hours to 0.1% Creep	% Elongation	% Reduction Area
TRW-NASA VIA Modification IV	954	1750	106.8	30	26.5	5.8	4.6	6.6
	954	1750	106.8	30	20.6	8.4	4.2	0.8
	954	1750	106.8	30	19.6	2.5	3.1	1.9
	830	1525	482.6	70	77.6	6.9	3.1	2.0
	830	1525	482.6	70	58.1	8.0	3.5	1.9
	760	1400	620.5	90	345.6	13.5	2.7	2.5
					290.6	10.2	2.0	1.6
TRW-NASA VIA Modification V	954	1750	106.8	30	20.1	3.4	5.6	2.4
	954	1750	206.8	30	15.3	6.0	6.0	2.3
	954	1750	206.8	30	(2)			
	830	1525	482.6	70	20.9	4.1	2.4	3.5
	830	1525	482.6	70	18.0	3.5	1.9	2.0
	760	1400	620.5	90	280.3	6.1	2.7	2.5
	760	1400	620.5	90	180.3	4.9	2.0	1.6
AFML Goal						30		

(1) Processing included HIP consolidation at 1316°C (2400°F)/103.4 MPa (15 Ksi)/4 hours plus heat treatment of 899°C (1650°F)/24 hours/air cool.

(2) Specimen failed on loading.

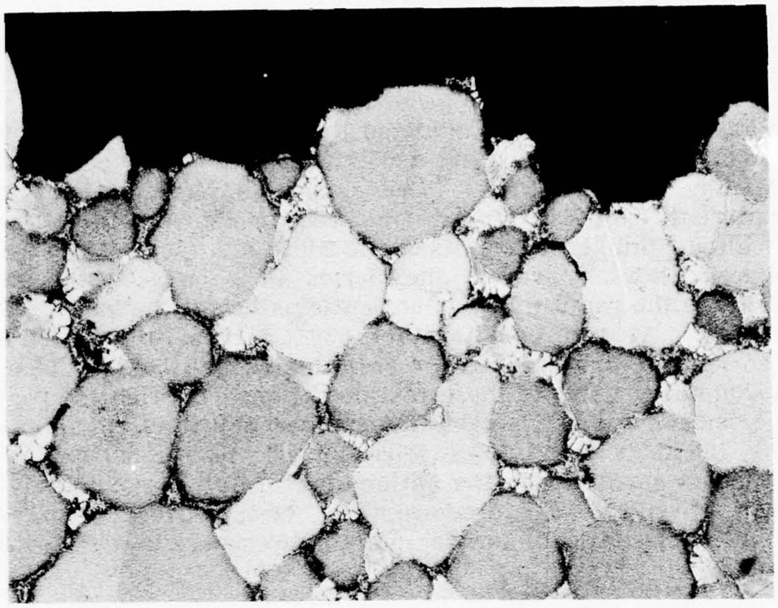
The additional creep-rupture tests conducted at 830°C (1525°F) and 760°C (1400°F) indicated that the high hafnium alloy, Modification IV, was superior in terms of rupture life and time to reach 0.1% creep compared to the Modification V alloy. At 830°C (1525°F), for example, Modification IV alloy exhibited an average rupture life of 67.9 hours compared to the 19.5 hour average for the Modification V alloy. Unlike the results of the additional tensile tests conducted on the powder metallurgy alloys, however, the Series III compositions as a group were relatively inferior to the rupture life values anticipated for the base TRW-NASA VIA in the cast condition. At test conditions of 830°C (1525°F)/482.6 MPa (70 ksi), for example, the anticipated rupture life for cast-to-size test bars of TRW-NASA VIA is approximately 200 hours (29). At these test conditions, Modification IV, the best of the Series III powder metallurgy alloys exhibited an average life of only approximately 67.9 hours. At the 760°C (1400°F)/620.4 MPa (90 ksi) test condition, Modification IV averaged 318.6 hours compared to the anticipated life of approximately 600 hours for cast TRW-NASA VIA (29). The creep-rupture results overall thus suggested little potential for the applicability of powder metallurgy superalloys as isothermal forging die materials.

(e) Metallographic Analysis

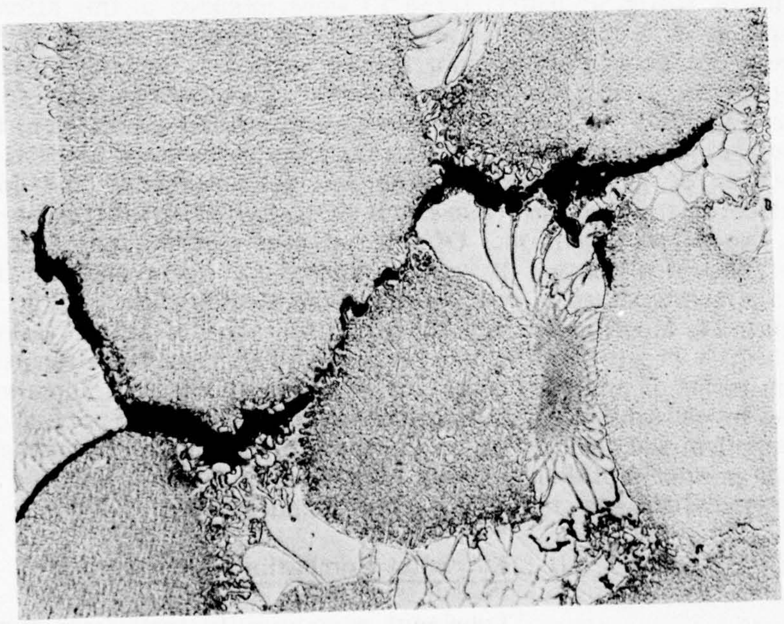
Metallographic analysis was conducted upon failed test bar specimens of the two Series III alloys evaluated in both the tensile and the creep-rupture tests. Only those tensile specimens tested at 927°C (1700°F) and those creep-rupture specimens tested at 954°C (1750°F) were included in this effort to provide a direct comparison with the Series I and Series II powder metallurgy results. The analyses indicated that an intergranular failure mode was common to both the tensile and the creep-rupture tests. A typical example of an intergranular fracture path is shown in Figure 15 for a 954°C (1750°F) creep-rupture test specimen of Modification V alloy. For both of the Series III alloys the fracture paths were associated with large, eutectic colonies of gamma-prime. The microstructures of the failed test bar specimens reflected the fact that there was little significant difference in properties between the two Series III alloys. Of interest also was the fact that the chemistry alterations of the Modification IV and V alloys did not result in any substantial differences in microstructure in comparison to the Modification II alloy, Figure 10. As shown in Figure 14, Modification II alloy exhibited slightly better yield strength and time to reach 0.1% creep than did the Series III alloys, but these results could not be rationalized in terms of microstructural differences. It was also apparent that oxygen content at the levels exhibited by the Modification II alloy (110 ppm) and the Series III alloys (both less than 100 ppm) did not play a significant role in the mechanical property response of the powder metallurgy alloys. Modification II alloy exhibited slightly better properties in spite of the fact that it contained higher oxygen.

(d) Lubricant Compatibility Tests

Lubricant compatibility tests were conducted on the Series III powder metallurgy alloys using OPT 112 and Deltaglaze 69 isothermal forging lubricants. Testing included 80 hours total exposure at each of three temperatures including 871°C (1600°F), 927°C (1700°F) and 982°C (1800°F). Evaluation included a metallographic examination of the type of surface attack and involved measurements of



(a) 100X



(b) 500X

Figure 15. Light photomicrographs of Task I (powder metallurgy) Series III TRW-NASA VIA Modification V alloy showing intergranular fracture mode typical of creep rupture test specimens.

the depth of penetration of the reaction products. The results of these lubricant compatibility tests are shown in Table X in terms of the average depth of attack along the surface. The results indicated that for the Series III alloys the Deltaglaze 69 forging lubricant was somewhat more aggressive than the OPT 112 in its attack on the modified TRW-NASA VIA powder metallurgy alloys. The mode of attack of the forging lubricants was in the form of a generalized surface type corrosion rather than a localized penetration along grain boundaries or remnants of prior particle boundaries. Examples of the typical appearance of this generalized corrosion attack is shown in Figure 16 for the Modification IV and V alloys after 80 hours exposure at 982°C (1800°F) to the OPT 112 forging lubricant. One feature apparent in the photomicrograph of the Modification IV alloy, Figure 16a, is the rather uneven nature of the attack along the surface. This effect was characteristic of both compositions at both 927°C (1700°F) and 982°C (1800°F). This uneven penetration was not associated with grain boundary areas or any other apparent discontinuities in the microstructure of the alloy. Although the depth of penetration was somewhat greater for the Modification IV alloy in terms of actual measured values, there appeared to be little significant difference between the two alloys in their overall resistance to attack by either of the forging lubricants. This may have reflected, in part, their similarity in compositions.

(e) Thermal Fatigue/Creep Interaction Test Results

Thermal fatigue/creep interaction tests were conducted on two specimens from each of the Series III alloys. The specimens were loaded at 927°C (1700°F)/172 MPa (25 ksi) and the test cycle ran for four hours under load and temperature after which specimens were unloaded and cooled at the same time to room temperature for 5-10 minutes and then reheated and loaded for the next cycle. Failure consisted of complete specimen separation. The results of these thermal fatigue tests are listed in Table XI. The data indicate that both of the Series III powder metallurgy superalloys exhibited extremely poor thermal fatigue resistance. Neither alloy exhibited failure lives exceeding one cycle before complete specimen separation occurred. Metallographic analysis was conducted on the failed fatigue test specimens and indicated that an intergranular failure mode, similar to that for the tensile and creep rupture test specimens, was observed for both alloys, and there was little difference between the various test specimens. The poor thermal fatigue resistance of the Series III powder metallurgy alloys was difficult to rationalize on the basis of the creep rupture properties exhibited at other temperatures. At 954°C (1750°F)/206.8 MPa (30 ksi), for example, the rupture lives for Modifications IV and V alloys ranged from 15.3-26.5 hours. In thermal fatigue at 927°C (1700°F)/172 MPa (25 ksi), however, none of the specimens exceeded a single cycle (or withstood more than four hours at the applied load). Metallographic analysis indicated that there was nothing obvious in the microstructures of the alloys to explain this poor thermal fatigue resistance.

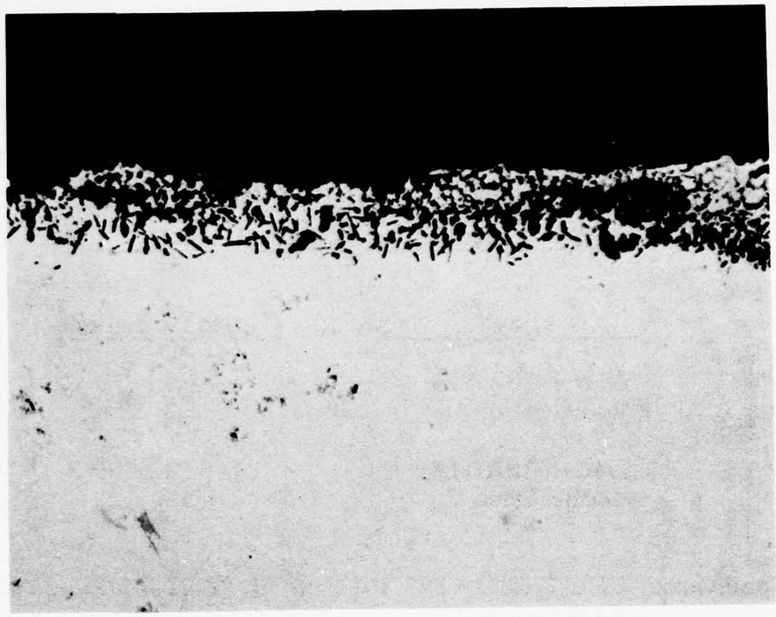
Table X

Lubricant Compatibility Test Results for Task I

(Powder Metallurgy) Series III Alloys[1]

OPT 112 Lubricant	871°C (1600°F) mm	871°C (1600°F) inch	927°C (1700°F) mm	927°C (1700°F) inch	982°C (1800°F) mm	982°C (1800°F) inch
TRW-NASA VIA Mod IV	.00381	.00015	.01753	.00069	.02489	.00098
TRW-NASA VIA Mod V	.00229	.00009	.01854	.00073	.02210	.00087
Deltaglaze 69 Lubricant						
TRW-NASA VIA Mod IV	.00813	.00032	.01651	.00065	.06375	.00251
TRW-NASA VIA Mod V	.00483	.00019	.01473	.00058	.02616	.00103

(1) Measurements indicate depth of penetration after 80 hours exposure at the various test temperatures.



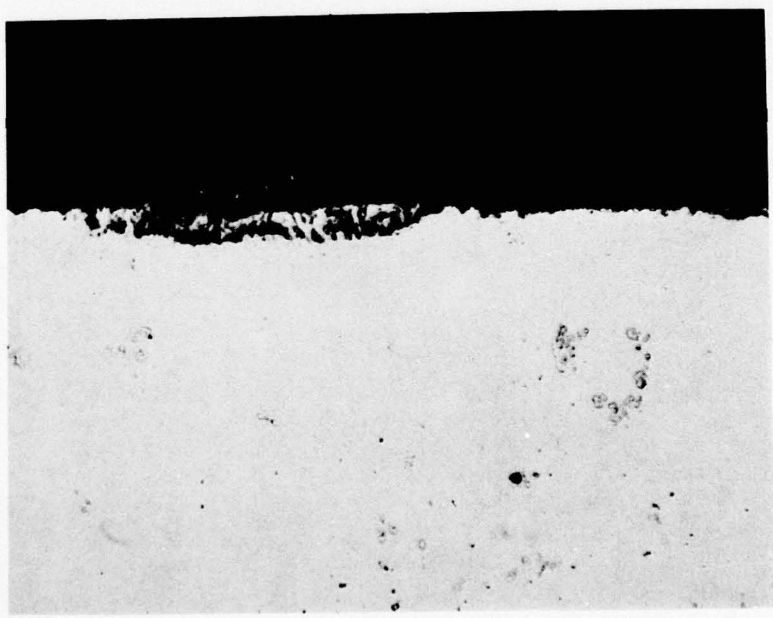


Figure 16. Light photomicrographs of Task I (powder metallurgy) Series III alloys after 80 hours lubricant compatibility test with OPT 112 forging lubricant at 982°C (1800°F). Unetched condition. Magnification, 100X.

Table XI

Thermal Fatigue/Creep Interaction Test Results for Task I (Powder Metallurgy) Series III Alloys[1]

Alloy[2]	Cycles to Failure
TRW-NASA VIA Modification IV	1
	1
TRW-NASA VIA Modification V	1
	1

(1) Test Conditions: 25°C (70°F) - 927°C (1700°F), 0-172.4 MPa (25 Ksi), 4 hour cycle.

(1) Processing conditions for all specimens included HIP at 1316°C (2400°F)/103 MPa (15 Ksi)/4 hours plus 899°C (1650°F)/24 hours/air cool heat treatment.

B. Task II - Casting Superalloys

The Task II studies were directed towards the evaluation of nine candidate alloy compositions compatible with current casting technology. The nine alloys were divided into three series with four alloys in Series I, three in Series II and two in Series III. The following sections contain descriptions of the procedures used for the development of these casting nickel-base superalloys as well as a discussion of the results of the alloy development effort.

1. Experimental Procedures

a. Melting and Casting

The casting alloys were prepared at the TRW Experimental Foundry in Minerva, Ohio, in the form of 22.6 kg (50 pound) 8.9-cm (3.5-inches) x 8.9-cm (3.5-inches) x 17.8-cm (7 inches)long rectangular blocks of virgin material. For the Series I and II alloys one block was made of each of the alloys using current vacuum precision investment casting techniques. For the more extensive mechanical property evaluations of Series III, two blocks were required for each alloy. From work conducted at TRW under internal sponsorship (49) it was anticipated that the section size effects present in these castings would be similar to those obtained in the larger forging die blocks required for the Phase II fabrication effort. For all of the castings a superheat temperature 1649^{o}C (3000^{o}F) and a pour temperature of 1510^{o}C (2750^{o}F) were used. The total cycle time from power-on to final pour was approximately 45-60 minutes for each heat. The Series I castings were invested as shown in the schematic illustration of Figure 17a. The molds were preheated at 982^{o}C (1800^{o}F) for 3 hours and were packed in MgO grain. This procedure resulted in the Series I castings solidifying from the outside inward to the center of the casting and created some center-line porosity. To eliminate this condition the molds for the Series II and Series III castings were invested as shown in the schematic illustration of Figure 17b. The pour cup portion of the casting was wrapped with two layers of Kaowool and then set on top of a base of steel shot lining the bottom of the investment can. The steel shot was then added to approximately 5.1 cm (2 inches) farther up the mold. The rest of the can was then filled with MgO grain as usual. This resulted in a change in thermal gradient from the outside-inside situation prevalent for the Series I castings to a bottom-top gradient. The steel shot conducted the heat away from the bottom of the casting and allowed it to solidify first while the Kaowool insulated the top allowing it to remain molten for a longer period of time and thus localizing porosity at the very top and in the pour cup portion of the casting.

b. Mechanical Property Evaluations

The mechanical property evaluations were similar to those conducted on the Task I powder metallurgy alloys. Mechanical property screening studies including tensile and creep-rupture tests were conducted on Series I and II alloys on specimens machined from the corner portions of the cast die blocks. As a check, tests were also conducted on specimens machined from the center portion of the block to determine section size effects. To accomplish this cylindrical machining preforms were first EDM machined from center and corner locations as shown for a typical cast block in Figure 18. Final machining operations were then performed on these preforms.

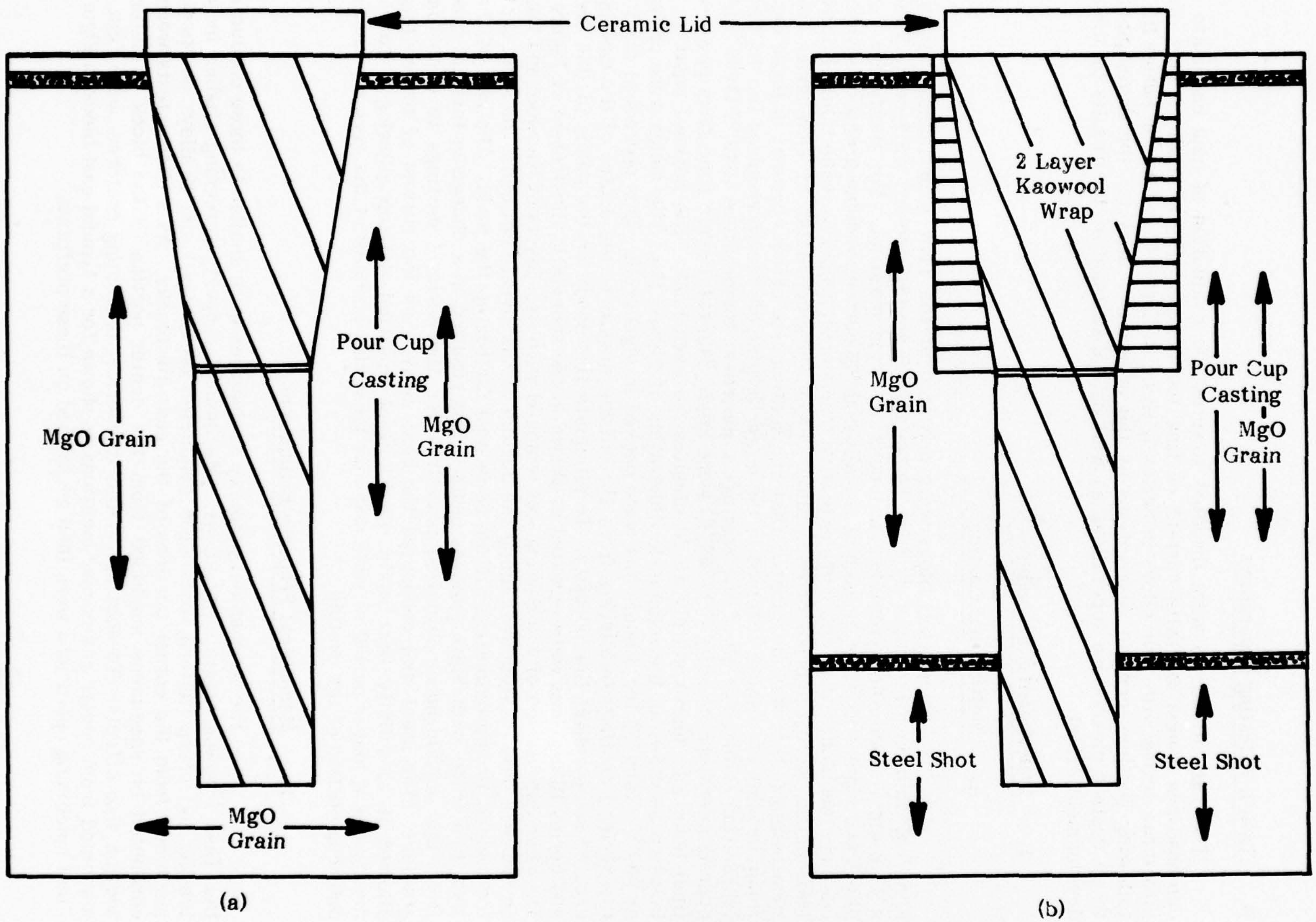


Figure 17. Mold investment scheme for Task II (casting superalloys) for (a) Series I and (b) Series II and III castings.

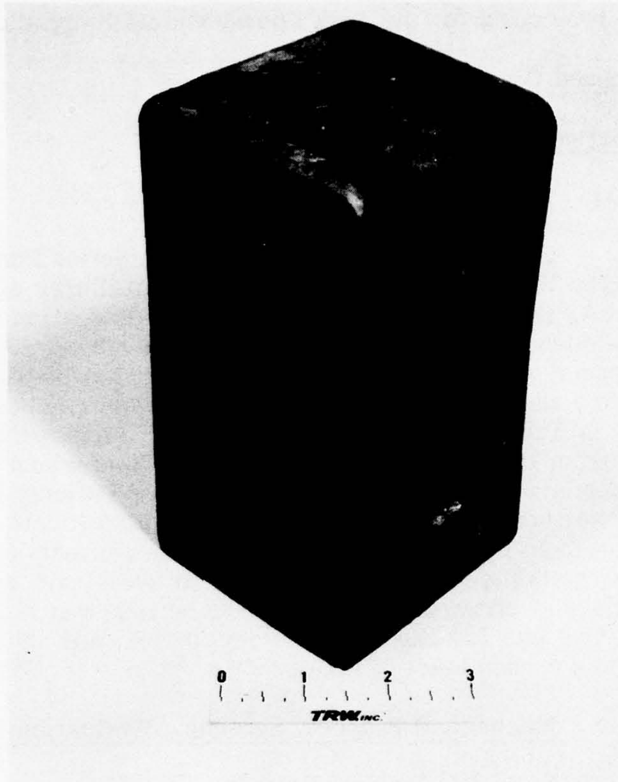


Figure 18. Cast block of Task II (casting superalloy) alloy showing location of specimens taken for mechanical property evaluations.

The more extensive mechanical property characterizations conducted on the two Series III alloys were also done on specimens machined from the corner and center portions of the cast blocks. This included tensile, creep-rupture, lubricant compatibility and thermal fatigue-creep interaction tests employing procedures identical to those described previously for the Task I powder metallurgy alloys.

2. Results and Discussion

a. Series I Alloy Studies

(1) Alloy Formulation

The four alloys selected for the Series I casting alloy development work were similar to those for the Task I powder metallurgy effort and included TRW-NASA VIA, TAZ-8A, IN-792 and IN-100. With the exception of carbon content, the compositions were identical to those of the powder alloys. All of the casting alloys contained their normal amount of carbon. Similar to the powder metallurgy version of VIA, the casting alloy also contained no rhenium. The aim compositions of the four Series I alloys are listed in Table XII along with the actual compositions in weight percent of material removed from the cast rectangular blocks. Comparison of the aim compositions with the actual chemistries indicated that, with few exceptions, the desired compositions were achieved. Two such exceptions were the zirconium levels for the TRW-NASA VIA and the IN-792 alloys. In both instances the zirconium contents in the cast blocks were at approximately one half the desired level. These were not considered to be serious deviations from the aim chemistries. A third exception was the high carbon content of the IN-792 alloy. The aim for this material was 0.12%, and the actual chemistry of the cast block indicated a carbon level of 0.20%.

(2) Mechanical Property Screening Evaluation

(a) Tensile Test Results

Tensile tests were conducted at $927^{o}C$ ($1700^{o}F$) on three specimens from each of the Series I casting superalloys. The results of these tests are listed in Table XIII. The testing included duplicate tests on material machined from the centers of the cast blocks and a single test on material from the corners of the cast blocks. Specimens were evaluated from these various locations in order to determine the section size effect in these cast blocks. All specimens were evaluated in the as-cast condition. For comparative purposes data have also been listed in Table XIII for each alloy representative of the standard 0.6-cm (0.252-inch) diameter aircraft cast to size test specimens usually used in the development of most of these alloys. Because of the section size effect observed in nickel-base superalloys these properties would represent the optimum level possible for the particular compositions.

Table XII

Task II (Casting Superalloy) Series I Alloy Compositions in Weight Percent

Alloy		C	Cr	Mo	Ti	Al	Co	W	Zr	B	Ta	Cb	Hf	Other
TRW-NASA VIA	Aim	0.13	6.1	2.0	1.0	5.4	7.5	5.8	0.13	0.02	9.0	0.5	0.43	
	Actual	0.11	5.88	1.9	0.95	5.32	7.78	5.82	0.067	0.017	9.3	0.56	0.46	
TAZ-8A	Aim	0.13	6.0	4.0		6.0		4.0	1.0	0.004	8.0	2.5		
	Actual	0.14	6.29	3.99		5.89		4.15	0.99	0.007	8.03	2.58		
IN-792	Aim	0.12	12.4	1.9	4.5	3.1	9.0	3.8	0.10	0.02	3.9			
	Actual	0.20	12.4	1.89	4.15	3.04	9.14	3.72	0.058	0.021	3.85			
IN-100	Aim	0.18	10.0	3.0	4.7	5.5	15.0		0.06	0.014				1.0V
	Actual	0.17	9.68	3.01	4.74	5.54	14.86		0.044	0.017				0.99V

Table XIII

Task II (Casting Superalloys) Series I 927°C (1700°F) Tensile Test Results

Alloy	Location	Ultimate Tensile Strength MPa	Ultimate Tensile Strength Ksi	0.2% Offset Yield Strength MPa	0.2% Offset Yield Strength Ksi	% Elongation	% Reduction Area
TRW-NASA VIA	(1)	744.6	108	648.1	94	3.3	N.A.
	(2)	621.2	90.1	524.7	76.1	3.3	6.6
	(3)	546.1	79.2	499.2	72.4	1.1	1.2
	(3)	589.6	85.5	506.1	73.4	2.5	2.7
TAZ-8A	(1)	703.3	102		65.3	5.1	N.A.
	(2)	585.4	84.9	450.3	65.3	2.4	3.1
	(3)	574.4	83.3	418.5	60.7	2.2	3.1
	(3)	572.3	83.0	426.8	61.9	2.8	2.7
IN-792	(1)	689.5	100	475.7	69	10.3	N.A.
	(2)	472.9	68.6	339.9	49.3	11.1	16.6
	(3)	462.6	67.1	342.6	49.7	6.1	8.9
	(3)	452.3	65.6	357.1	51.8	3.3	8.1
IN-100	(1)	744.6	108	558.5	81	6.1	N.A.
	(2)	568.9	82.5	442.7	64.2	2.8	2.4
	(3)	570.0	82.8	456.5	66.2	3.5	4.3
	(3)	540.6	78.4	412.4	59.8	1.7	2.3

Air Force Program Goal: 517 MPa (75 Ksi) 0.2% offset yield strength at 927°C (1700°F) in a section size of 2.8 cm (7 inches).

(1) Cast to size 0.6 cm (0.252 inch) diameter aircraft test specimens. Data available in literature from the following sources:

TRW-NASA VIA - Simons, W.F., DMIC Memorandum 255, June 1971
TAZ-8A - Waters, W.J. and Freche, J.C., NASA TN D-3597, September 1966
IN-792 - Sims, C.T. and Hagel, W.C. eds., The Superalloys, John Wiley & Sons, 1972
IN-100 - Data Brochure from International Nickel, 1977

(2) Machined from corner of cast block.

(3) Machined from center of cast block.

The 927°C (1700°F) tensile data presented in Table XIII indicate a strong section size effect. For each alloy, the ultimate tensile and 0.2% yield strength values were significantly lower than those expected for cast-to-size test specimens 0.6 cm (0.252 inch) in diameter. This section size effect is a strong function of the casting cooling rate, a casting parameter which has been shown previously to exert a powerful effect on nickel-base superalloy mechanical property levels (50). The slower cooling rates prevalent in thick sections usually result in coarser gamma-prime precipitates and correspondingly lower strength properties. Note the improvement in properties between the corner specimens (fast cooling rate) and the center specimens (slower cooling rate).

The tensile results indicated that the TRW-NASA VIA alloy exhibited the highest ultimate tensile and 0.2% offset yield strengths of the four casting alloys. In spite of the strong section size effect, this alloy evidenced strong potential to meet the program yield strength goal. This alloy exhibited a 524.7 MPa (76.1 ksi) yield strength (exceeding the program goal) at the corner and approximately a 503.3 MPa (73 ksi) yield strength in the center of the cast block. The TAZ-8A and the IN-100, while exhibiting comparable tensile strength levels, were approximately 69-103 MPa (10-15 ksi) below the TRW-NASA VIA alloy in terms of yield strength. The IN-792 alloy exhibited the lowest tensile properties of the four Series I casting alloys. It was thus concluded that the TRW-NASA VIA alloy offered the most promising base composition to which modifications could be made in Series II to attain the yield strength program goal.

(b) Creep-Rupture Test Results

Creep-rupture tests were conducted at 954°C (1750°F)/206.8 MPa (30 ksi) on five specimens from each of the Series I alloys. The results of these creep-rupture tests are listed in Table XIV. For each of the alloys two of the test specimens were machined from the corner locations of the cast blocks while three specimens came from the central portions of the blocks. All specimens were tested in the as-cast condition. For comparative purposes data have also been listed for each alloy representative of standard 0.6-cm (0.252-inch) aircraft cast-to-size test specimens. Similar to the tensile results a strong section size effect was observed in the creep-rupture properties as indicated by the reduced rupture lives compared to values anticipated for cast to size test bars. This reduction in rupture life compared to anticipated levels was most pronounced in the TAZ-8A and IN-792 alloys while being significantly less pronounced in the TRW-NASA VIA and the IN-100 alloys. In general, however, the section size effect resulting from variations in specimen location was not as pronounced in the creep-rupture results as it was for the tensile properties. The effect of specimen location was manifested most strongly in IN-100 and had significantly less of an effect in the remaining alloys. For example, specimens machined from the corners of the IN-100 casting exhibited lives of approximately 130 hours, whereas the lives of specimens machined from the center of the casting ranged from 65.7 to 98.8 hours. This may be contrasted with the behavior of the IN-792 alloy in which the longest life (33.4 hours) was obtained from a specimen from the center of the casting. Lives of corner specimens varied from 16.3 to 31.1 hours for this alloy. The values for the times to reach 0.1% creep exhibited even less sensitivity to specimen location than did the rupture lives for the various alloys. None of the specimen locations could be differentiated in terms of trends in the times to reach 0.1% creep.

Table XIV

Task II (Casting Superalloys) Series I 954°C (1750°F)/206.8 MPa (30 Ksi)

Creep Rupture Test Results

Alloy	Location	Hours to Failure	Hours to 0.1% Creep	% Elongation	% R.A.
TRW-NASA VIA	(1)	420.0		-	-
	(2)	187.6	2.04	5.5	9.6
	(2)	150.0	1.13	4.7	5.5
	(3)	157.2	1.48	5.1	4.3
	(3)	75.2	1.27	5.1	4.7
	(3)	9.4	1.11	1.7	1.6
TAZ-8A	(1)	60	-	-	-
	(2)	14.7	0.07	4.1	2.8
	(2)	13.1	0.10	3.7	3.5
	(3)	18.9	0.36	3.3	3.2
	(3)	16.6	0.22	2.9	2.8
	(3)	25.7	0.24	2.3	0.8
IN-792	(1)	160.0	-	-	-
	(2)	16.3*	-	10.5	17.3
	(2)	31.1	0.06	7.9	17.4
	(3)	33.4	0.05	10.6	14.1
	(3)	1.3	0.66	2.0	0.8
	(3)	22.3	0.02	6.5	8.5
IN-100	(1)	160.0	-	-	-
	(2)	129.8	4.38	9.9	8.5
	(2)	129.1	1.59	7.2	9.6
	(3)	65.7	2.42	3.7	2.0
	(3)	97.3	3.99	7.7	10.8
	(3)	98.8	1.99	7.3	11.1
AFML Program Goal		-	30.0	-	-

(1) Cast to size 0.6 cm (0.252 inch) diameter aircraft test specimens. Data available in literature from the following sources:

TRW-NASA VIA
TAZ-8A
IN-792
IN-100

Referenced in Table XIII

(2) Machined from corner of cast block.

(3) Machined from center of cast block.

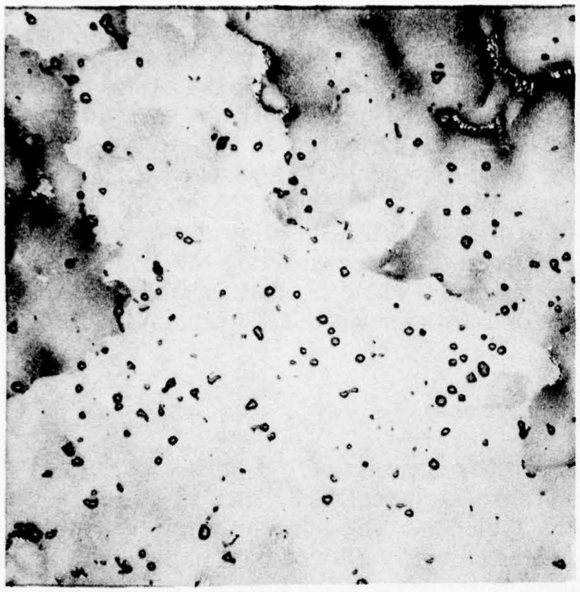
* Controller malfunction.

A comparison of the data for the four Series I alloys indicated that TRW-NASA VIA and IN-100 were clearly superior to the TAZ-8A and the IN-792 alloys. TRW-NASA VIA and IN-100 exhibited lives to 0.1% creep of 1.11 to 2.04 and 1.59 to 4.38 hours, respectively. All specimens of TAZ-8A and IN-792 reached 0.1% creep in less than 0.66 hours. In spite of the superiority of the TRW-NASA VIA and IN-100 alloys to the other Series I alloys, however, the life of 0.1% creep offered by these compositions was considerably below the program goal of 30 hours. These relatively low lives exhibited by the TRW-NASA VIA alloy and the currently used IN-100 isothermal forging die material underscored the need for alloy development efforts to improve the dimensional stability of forging tooling.

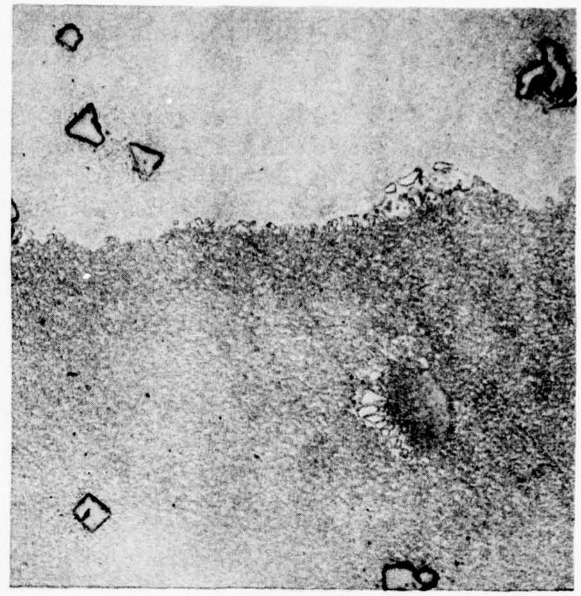
(c) Metallographic Analysis

Typical microstructures for two of the Series I alloys in the as-cast condition are shown in Figure 19. These include IN-792 and IN-100 superalloy specimens from the corner portions of the cast blocks. The photomicrographs illustrate the complexity of the structures developed in these highly alloyed cast nickel-base alloys. An important feature of these microstructures is the presence of large primary gamma-prime eutectic colonies both within grains and along grain boundaries. Previous work on these alloy systems has shown that the massive gamma-prime regions are not just large globules of primary gamma-prime but are a much finer dispersion within the colonies (51). To a certain extent, these gamma-prime formations have been shown to be beneficial to the elevated temperature creep-rupture properties of nickel-base superalloys. Alloy compositions containing relatively little of this microconstituent exhibit inferior creep rupture properties compared to alloys characterized by larger amounts of the primary gamma-prime formation.

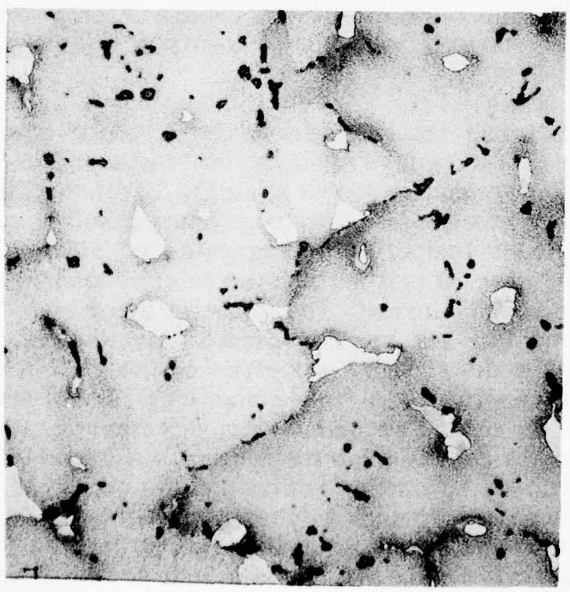
Examination of failed test bar specimens indicated that an intergranular failure mode was common to all four alloys for both the tensile and creep-rupture tests. Examples of such intergranular cracks are shown in Figure 20 for the TRW-NASA VIA and the TAZ-8A alloys. While the presence of massive primary gamma-prime eutectic formations is beneficial to the high temperature properties of nickel-base superalloys, the photomicrographs shown in Figure 20 suggest that the relative amount and size of the colonies also may effect the property levels. The eutectic colonies in the TRW-NASA VIA, for example, are much smaller and more dispersed than those in the TAZ-8A alloy. This may account, in part, for the superior properties of the TRW-NASA VIA alloy compared to TAZ-8A. A similar observation was made concerning gamma-prime eutectic formation during the analysis of specimens prepared from different locations within the cast rectangular blocks. Examples of these microstructural differences are shown in Figure 21 for TRW-NASA VIA specimens from the corner and the center of the cast block. The microconstituents in the corner specimen (characterized by a faster casting cooling rate) appear somewhat finer in size than those in the microstructure of the center specimen. This includes the carbides within the structure as well as the gamma-prime formations. Note also the difference in intragranular gamma-prime size as shown in the higher magnification Scanning Electron Microscope photomicrographs. The intragranular gamma-prime precipitates characteristic of the corner specimen, Figure 21b, are much finer in size than those seen in the center specimen, Figure 21d. These microstructural differences resulting from variations in casting cooling rate have been reported in other nickel-base superalloys and are responsible for the section size effect observed in the mechanical property response (50).



(a) IN-792, 100X



(b) IN-792, 500X



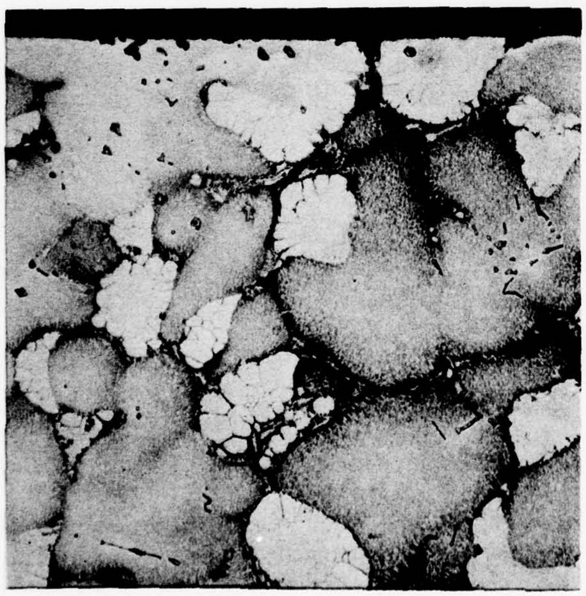
(c) IN-100, 100X

(d) IN-100, 500X

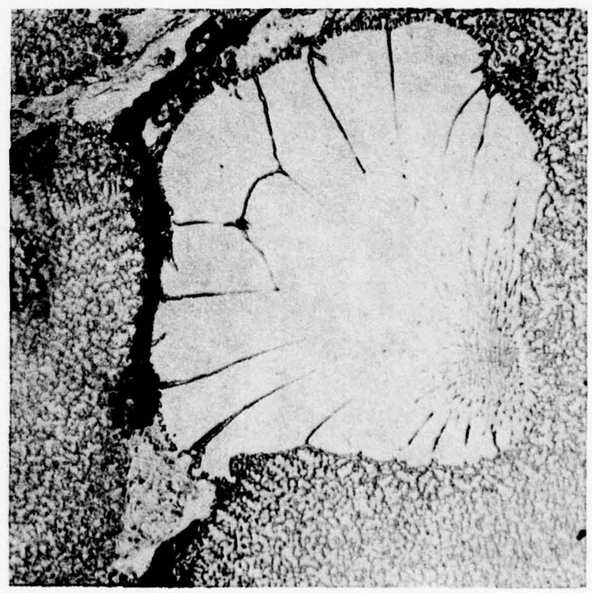
Figure 19. Light photomicrographs of Task II (casting superalloys) Series I alloys IN-792 and IN-100 as-cast microstructures. Specimens were prepared from the corners of the cast blocks.

(a) TRW-NASA VIA, 100X

(b) TRW-NASA VIA, 500X

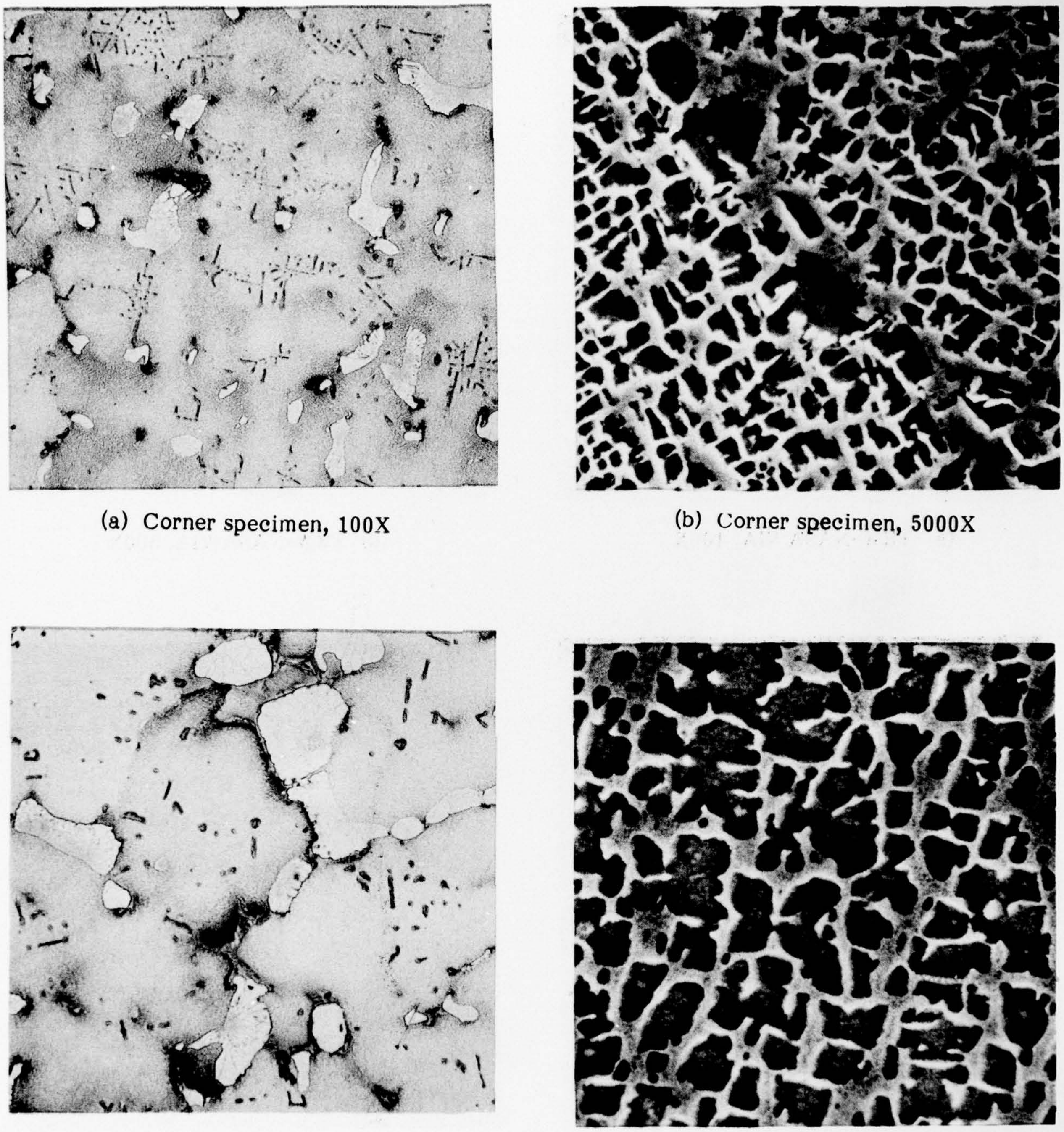


(c) TAZ-8A, 100X



(d) TAZ-8A, 500X

Figure 20. Light photomicrographs of Task II (casting superalloy) Series I TRW-NASA VIA and TAZ-8A alloys showing typical examples of intergranular cracking in creep-rupture test specimens.



(a) Corner specimen, 100X

(b) Corner specimen, 5000X

(c) Center specimen, 100X

(d) Center specimen, 5000X

Figure 21. Light and SEM photomicrographs of Task II (casting alloy) Series I TRW-NASA VIA alloy showing section size effect between specimens from the corner and center of the cast block.

b. Series II Alloy Studies

(1) Alloy Formulation

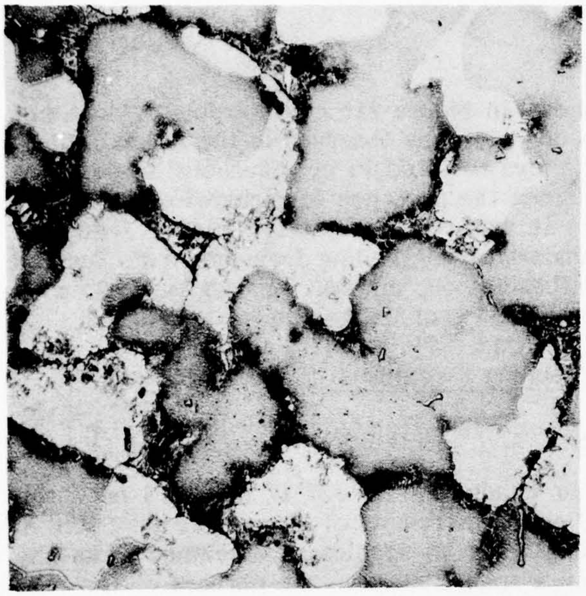
For the Task II casting superalloys, the three aim compositions presented in Table XV were selected for evaluation in Series II. The aim chemistries are presented in terms of weight percent. These alloys were all modifications of TRW-NASA VIA, chosen as the base alloy for Series II because it exhibited the best overall combination of tensile and creep-rupture properties in the Series I mechanical property screening studies. These properties included the highest ultimate tensile and yield strengths, the longest creep-rupture lives and times to reach 0.1% creep comparable to IN-100, which exhibited the next best creep-rupture properties. Because of the predominantly intergranular failure mode of the Series I TRW-NASA VIA test specimens,the alloy variations shown in Table XV were designed to enhance the creep-rupture properties pincipally through grain boundary strengthening. Compared to the base alloy composition shown in Table XII, Modification I was aimed at 2.0% hafnium, an increase over the base alloy level of 0.46%. Similar to the modifications made for the Task I powder metallurgy alloys, the hafnium content was increased in order to achieve the improvements in elevated temperature rupture life, ductility and tensile strength reported for cast TRW-NASA VIA at the higher hafnium levels (43). Reduced levels of carbon, boron, and zirconium, also reported to improve creep strength in nickel-base superalloys (45,46) were also included in this modification. Modification II was similar in aim composition to that of Modification I with the exception that boron and zirconium remained at their original levels and rhenium was added at an intended level of 0.22%. Rhenium was added to this composition because it has demonstrated the capability to enhance grain boundary strength (30) and also provides alloy strengthening in a number of advanced superalloy turbine blade compositions (47). Modification III alloy was aimed at a higher hafnium content (2.5%), with tantalum reduced from 9.0 to 6.0% as a means of adjusting the gamma-prime content in line with that of conventional TRW-NASA VIA. The Modification III composition was previously evaluated in terms of 982°C (1800°F) creep-rupture properties as part of a TRW internally funded program, with the result that an improved capability over conventional TRW-NASA VIA was demonstrated (52).

The actual compositions in weight percent of material removed from the cast rectangular blocks of the three Series II alloys are also listed in Table XV. Comparison of the aim compositions with the actual chemistries indicated that the desired composition for the Modification III alloy was achieved, but was not achieved for the Modification I and II alloys. For both of these alloys the actual hafnium level was approximately 0.5% below the desired 2.0% level. The alloys were not remelted because it was felt that the actual hafnium content of approximately 1.5% represented a high enough level to establish the effect of increased content of this element compared to the 0.46% level of the base composition. Typical microstructures of the Series II alloys in the as-cast condition are shown in Figure 22. These specimens were prepared from the center of the cast rectangular blocks. Comparison with the microstructure shown in Figure 21c for the Series I TRW-NASA VIA composition from a similar location in that casting indicates two major microstructural changes resulting from the chemistry modifications. First is the appearance of the primary gamma-prime eutectic colonies. These microconstituents appear more massive in the Series II alloys and are present in greater amounts than in the Series I alloy. Second is the reduction in the numbers of carbides

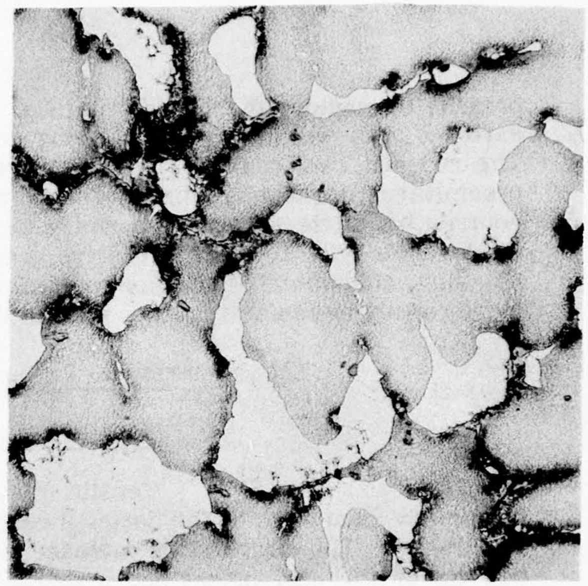
Table XV

Task II (Casting Superalloy) Series II Alloy Compositions in Weight Percent

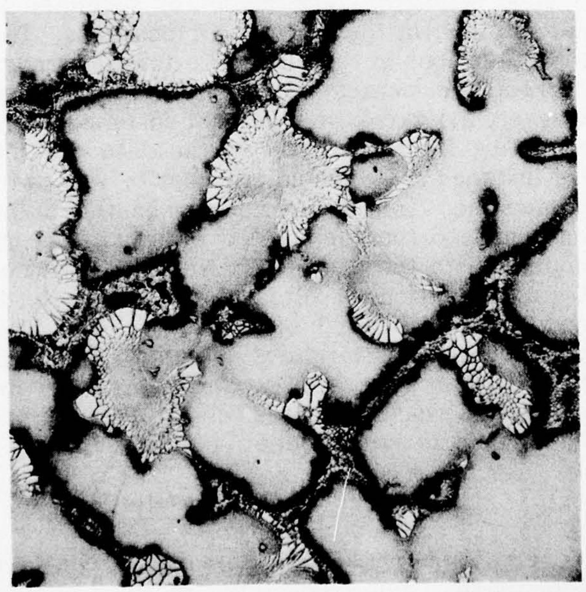
Alloy		C	Cr	Mo	Ti	Al	Co	W	Zr	B	Ta	Cb	Hf	Re
TRW-NASA VIA Modification I	Aim	0.1	6.1	2.0	1.0	6.0	7.5	5.8	.01	.01	9.0	0.5	2.0	
	Actual	0.085	6.0	1.86	.85	5.4	7.49	5.97	.005	.009	10.43	0.42	1.5	
TRW-NASA VIA Modification II	Aim	0.05	6.1	2.0	1.0	6.0	7.5	5.8	0.13	0.2	9.0	0.5	1.5	0.22
	Actual	0.037	5.82	1.9	.76	6.0	7.36	5.74	0.12	.019	9.8	0.43	1.63	0.22
TRW-NASA VIA Modification III	Aim	0.05	6.1	2.0	1.0	6.0	7.5	5.8	0.01	0.01	6.0	0.5	2.5	
	Actual	0.053	6.27	2.01	0.75	6.0	7.59	5.72	0.037	0.006	6.92	0.46	2.13	



(a) TRW-NASA VIA
Modification I



(b) TRW-NASA VIA
Modification II



(c) TRW-NASA VIA
Modification III

Figure 22. Light photomicrographs of Task II (casting superalloy) Series II alloys showing typical as-cast microstructure from center of cast block. All magnifications - 100X.

present throughout the microstructure. As shown in Figure 21c considerable evidence of carbides both within grains and along grain boundaries was observed in the Series I alloy. The reduced carbon content of the Series II alloys resulted in considerably less carbide precipitation throughout the microstructure. Since the presence of carbides within grains contributed little to grain boundary strength, it was reasoned that a carbon reduction would contribute to more effective grain boundary strengthening from elements such as titanium, columbium, tantalum and hafnium. These strong carbide formers would thus be more readily available to participate in the precipitation of the gamma-prime phases.

(2) Mechanical Property Screening Evaluation

(a) Tensile Test Results

Tensile tests were conducted at 927°C (1700°F) on three specimens from each of the Series II casting alloys. The results of these tests are listed in Table XVI. The evaluations included duplicate tests on specimens machined from the centers of the cast blocks and a single test on a specimen from the corners of the cast blocks. All specimens were tested in the as-cast condition. The tensile results indicated comparable strength levels for all three of the modifications. For test specimens machined from the corners of the cast blocks, the ultimate tensile strengths ranged from 623.3-630.9 MPa (90.4-91.5 ksi) while the yield strengths ranged from 488.2-528.2 MPa (70.8-76.6 ksi). The Modification II specimen was the only one to meet the program goal of 517 MPa (75 ksi) yield strength. The effect of specimen location was much more pronounced in these alloys than in the Series I compositions. This effect was manifested in terms of substantial reductions in both ultimate tensile strength and yield strength for specimens machined from the center portions of the castings. Examination of the fracture surfaces, however, indicated that in most instances microporosity was observed on the fractures, accounting for the scatter in the data and the relatively poor tensile results. In the single instance in which microporosity was not located on the fracture surface of a specimen machined from the center of a casting (a Modification III specimen), the results were comparable to those exhibited by specimens machined from corners of the cast blocks. Comparison to the Series I TRW-NASA VIA composition indicated that the Series II chemistry modifications resulted in approximately the same ultimate tensile strength, yield strength and ductility exhibited by the Series I alloy. A plot of the yield strength for the Series II casting superalloys compared to the Series I TRW-NASA VIA alloy is shown in Figure 23. All of the yield strength data values were included in this plot for each of the alloys. The increase in scatter due to the microporosity in the specimens from the center of the castings is quite evident in this plot.

(b) Creep-Rupture Test Results

Creep-rupture tests were conducted at 954°C (1750°F)/206.8 MPa (30 ksi) on five specimens from each of the Series II alloys and the results of these tests are listed in Table XVII. For each of the alloys three of the test specimens were machined from the corner locations of the cast blocks while two specimens came from the central portions of the blocks. All specimens were tested in the as-cast condition. Analysis of the creep-rupture results for specimens machined from the corners of the cast blocks indicated that Modification II, featuring a higher hafnium content as well as a

Table XVI

Task II (Casting Superalloys) Series II 927°C (1700°F) Tensile Test Results

Alloy	Location	Ultimate Tensile Strength MPa	Ultimate Tensile Strength Ksi	0.2% Offset Yield Strength MPa	0.2% Offset Yield Strength Ksi	% Elongation	% Reduction Area
TRW-NASA VIA Modification 1	(1)	626.1	90.8	507.5	73.6	2.7	-
	(2)	386.1	56.0(3)	379.2	56.0	1.0	-
	(2)	307.6	44.6(3)	307.6	44.6	1.2	2.8
TRW-NASA VIA Modification II	(1)	630.9	91.5	528.2	76.6	4.3	7.0
	(2)	285.5	41.4(3)	285.5	41.4	1.2	0.8
	(2)	373.0	54.1(3)	373.0	54.1	1.1	-
TRW-NASA VIA Modification III	(1)	623.3	90.4	488.2	70.8	3.6	4.7
	(2)	616.4	89.4	505.4	73.3	2.8	2.4
	(2)	469.5	68.1(3)	460.7	66.8	1.1	0.4

Air Force Program Goal - 517 MPa (75 Ksi) 0.2% offset yield strength at 927°C (1700°F) in a section size of 2.8 cm (7 inches)

(1) Machined from corner of cast block
(2) Machined from center of cast block
(3) Porosity observed on fracture surface

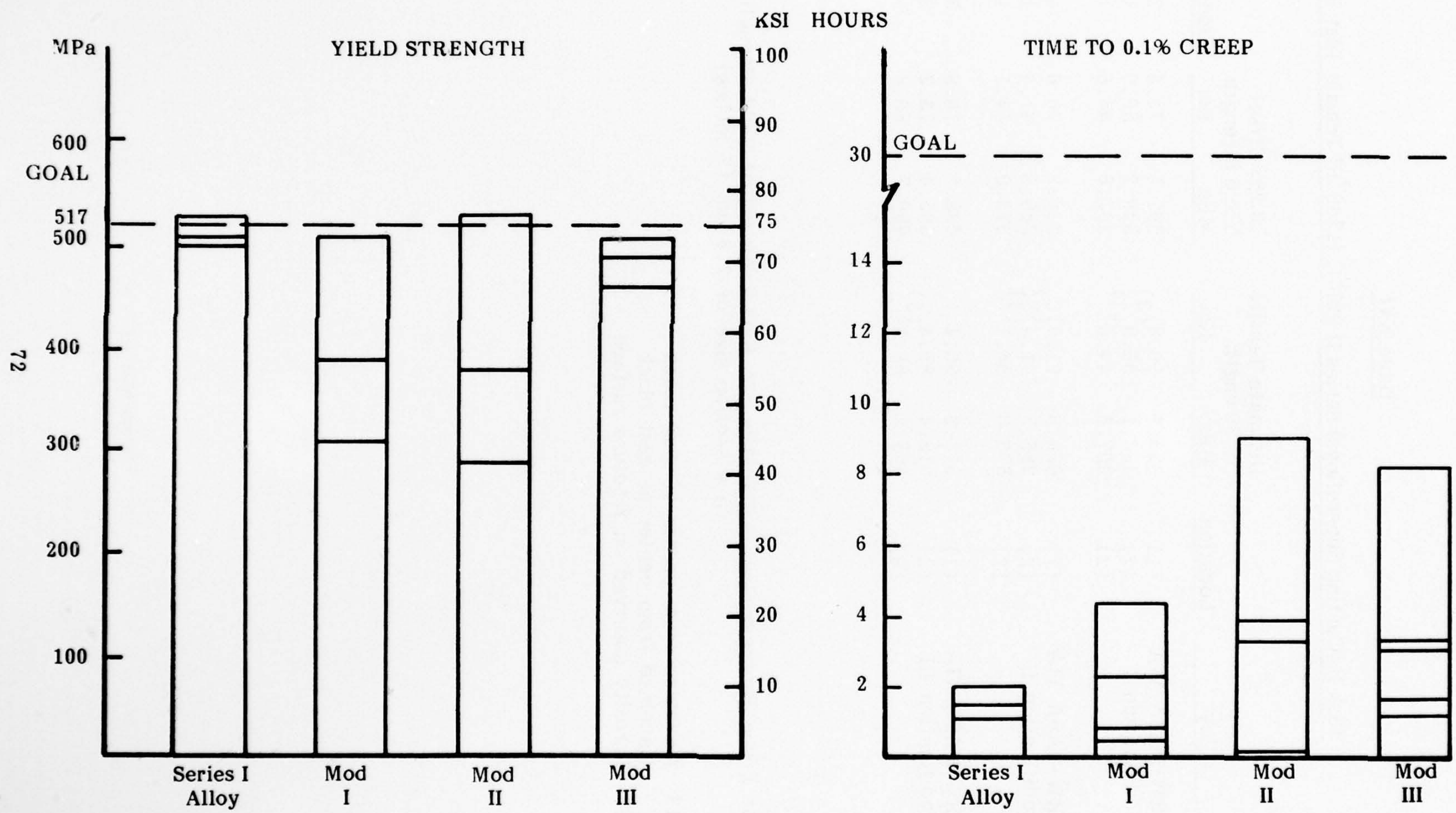


Figure 23. Yield strength and time to 0.1% creep for Task II (casting superalloy) Series II alloys compared to Series I TRW-NASA VIA alloy.

Table XVII

Task II (Casting Superalloys) Series II 954°C (1750°F)/206.8 MPa (30 Ksi)

Creep Rupture Test Results

Alloy	Location	Hours to Failure	Hours to 0.1% Creep	% Elongation	% Reduction Area
	(1)	59.4	4.37	1.7	2.4
TRW-NASA VIA	(1)	12.6	2.29	1.2	1.2
Modification I	(1)	51.6	0.52	1.6	0.8
	(2)	75.7	0.5	2.4	1.8
	(2)	1.1	0.83	0.9	1.5
	(1)	105.3	9.0	3.7	1.6
TRW-NASA VIA	(1)	83.6	3.86	1.9	0.4
Modification II	(1)	95.8	3.3	2.1	2.4
	(2)	1.55	0.24	1.3	0.4
	(2)	6.04	0.04	1.2	0.4
	(1)	87.0	1.7	3.0	3.5
TRW-NASA VIA	(1)	75.4	1.24	3.2	4.3
Modification III	(1)	67.0	3.11	2.1	2.7
	(2)	31.9	8.33	0.5	1.2
	(2)	73.0	3.39	2.8	1.9
AFML Program Goal			30.0		

(1) Machined from corner of cast block
(2) Machined from center of cast block

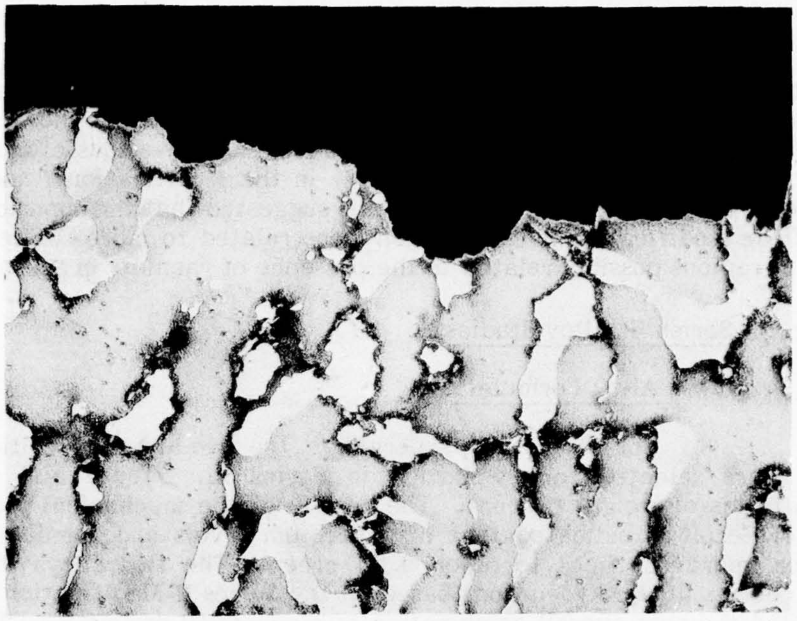
rhenium addition compared to the base alloy, exhibited the best overall creep-rupture properties of the Series II casting alloys. This alloy offered an average rupture life of 94.9 hours and times to reach 0.1% creep ranging from 3.3 to 9.0 hours. Specimens from the corner of the Modification III block exhibited the next best combination of creep-rupture properties. Modification III featured the highest hafnium content and exhibited an average rupture life of 76.4 hours and times to reach 0.1% creep ranging from 1.7 to 3.11 hours. Modification I alloy, similar in hafnium content to the Modification II alloy, but containing no rhenium exhibited an average rupture life of 41.2 hours and times to reach 0.1% creep ranging from 0.52 to 4.37 hours.

The effect of specimen location was somewhat inconsistent in the Series II alloys compared to the Series I compositions. For Modification I, for example, duplicate specimens machined from the centers of the cast blocks exhibited rupture lives of 75.7 and 1.1 hours, and times to reach 0.1% creep of 0.5 and 0.83 hours, respectively. The 75.7 hour rupture life was far in excess of the average of 41.2 hours exhibited by specimens machined from the corners of the cast block. The effect was more consistent for Modification II, in which center specimens exhibited rupture lives of 1.55 and 6.04 hours and times to reach 0.1% creep below 0.24 hours. Unlike the tensile specimens, microporosity was not observed on the fracture surfaces of these test specimens. Inconsistency was again apparent in the Modification III alloy in that one of the center specimens exhibited 8.33 hours to reach 0.1% creep, the second highest value observed for the Series II castings.

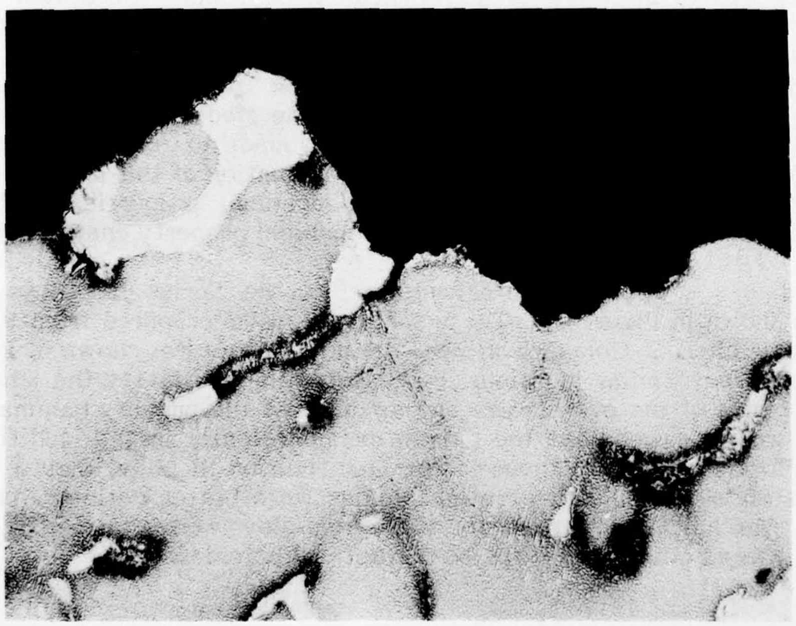
Comparison with the Series I TRW-NASA VIA composition indicated that the increased hafnium, reduced carbon, boron and zirconium and the addition of rhenium in the various Series II alloys resulted in improvements in the times to reach 0.1% creep for these compositions. These improvements are shown in Figure 23, which includes a plot of all the test values of time to reach 0.1% creep for the Series II alloys compared to the Series I base composition. The overall creep-rupture mechanical property trends were similar to those for the powder metallurgy alloys in that while the hafnium, boron, zirconium and rhenium chemistry modifications improved the times to reach 0.1% creep compared to the TRW-NASA VIA base composition, the creep-rupture failure times and the rupture ductilities were inferior for the Series II modified alloys. For the Series I specimens machined from the corners of the cast block, for example, an average creep-rupture life of 168.9 hours was obtained, while the best of the Series II alloys, Modification II, averaged only 94.9 hours to failure. Similar reductions were observed for the Series II rupture ductilities. These results suggested that while the grain boundaries were strengthened to effect such creep deformation mechanisms as grain boundary sliding, once initiated, cracks propagated rapidly to failure compared to the base composition alloy.

(c) Metallographic Analysis

Metallographic analysis conducted upon failed test bar specimens indicated that an intergranular failure mode was typical for all three alloys for both the tensile and the creep-rupture tests. Typical examples of intergranular fracture paths are shown in Figure 24 for creep-rupture test specimens of Modification II and III alloys. Note that for both alloys the fracture paths were associated with the large,



(a) TRW-NASA VIA
Modification II



(b) TRW-NASA VIA
Modification III

Figure 24. Light photomicrographs of Task II (casting superalloy) Series II alloys TRW-NASA VIA Modifications II and III showing the intergranular failure mode typical of creep-rupture test specimens. Magnification, 100X.

eutectic colonies of primary gamma-prime. The microstructures of these failed test bar specimens were similar to those exhibited by the as-cast material shown in Figure 22 in that little significant difference was observed between the various alloys. The gamma-prime eutectic colonies appear somewhat larger in the Modification I and III alloys, but otherwise the structures are quite similar. This suggested that the superior creep-rupture properties of the Modification II composition were related to more subtle changes in the grain boundary regions possibly related to the presence of rhenium in this alloy.

c. Series III Alloy Studies

(1) Alloy Formulation

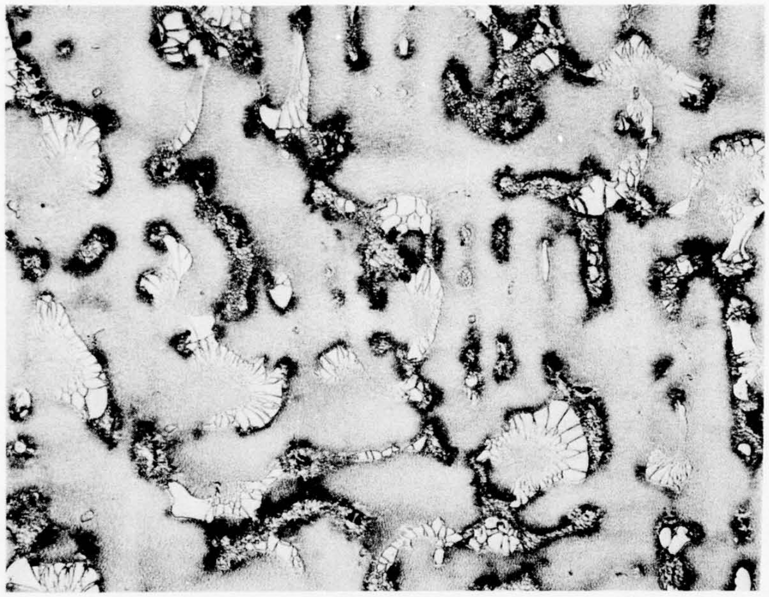
For the casting superalloys, the two aim compositions presented in Table XVIII were selected for evaluation in Series III. These aim chemistries are presented in terms of weight percent. The results of the mechanical property screening evaluations of Series II indicated that high hafnium levels and rhenium additions were beneficial for improved times to reach 0.1% creep. The two alloys thus selected for evaluation in Series III were variations based on the Series II Modification II and III alloys, which exhibited the best overall combination of mechanical properties. The Modification IV alloy was similar to the Series II Modification II composition, with the hafnium aim increased from 1.63 to 2.0%. Reduced boron, carbon and zirconium levels were also employed for this alloy, as they were in Series II Modification III. The Modification V composition was similar to the Series II Modification III alloy, with hafnium aimed at an increase from 2.13 to 2.5% and a 0.22% rhenium addition. The actual compositions in weight percent of material removed from the cast rectangular blocks of the two Series III alloys are also listed in Table XVIII. Comparison of the aim compositions with the actual chemistries indicated that the desired composition for the Modification IV alloy was achieved but that the actual hafnium content for the Modification V alloy was below the desired level (1.78% versus 2.5%). A decision was made to delay remelting of these cast blocks pending preliminary mechanical property evaluation of this composition. As will be discussed in the subsequent sections, the mechanical properties of this alloy were sufficiently promising to warrant complete mechanical property characterization.

Typical microstructures of the Series III alloys in the as-cast condition are shown in Figure 25. These specimens were prepared from the center of the cast rectangular blocks. Comparison with the microstructures shown in Figure 22 for the Series II alloys from similar locations in those castings indicates the similarity between the alloys in terms of the overall size and amounts of the primary gamma-prime eutectic colonies. It was observed that the gamma-prime colonies in the Modification IV alloy, Figure 25a, closely resembled those in the Modification III alloy, Figure 22c, in that the individual platelets within the eutectic colonies were better defined than those for the other alloys. The hafnium contents for these two alloys were above 2.0%, suggesting that high hafnium was associated with the better defined colonies.

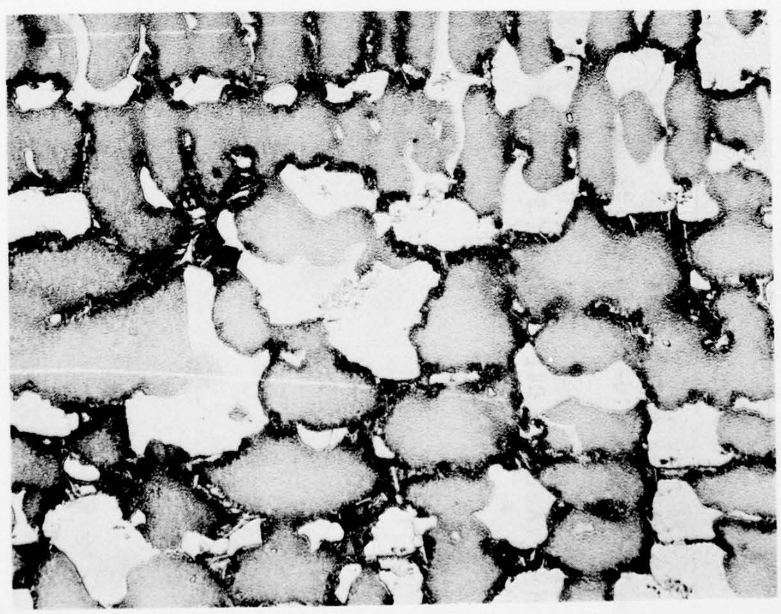
Table XVIII

Task II (Casting Superalloy) Series III Alloy Compositions in Weight Percent

Alloy		C	Cr	Mo	Ti	Al	Co	W	Zr	B	Ta	Cb	Hf	Re
TRW-NASA VIA	Aim	0.05	6.1	2.0	1.0	5.6	7.5	5.8	0.01	0.01	9.0	0.5	2.0	0.22
Modification IV	Actual	0.032	5.67	1.97	0.98	5.56	7.24	5.98	0.06	0.014	0.63	2.1	0.21	
TRW-NASA VIA	Aim	0.05	6.1	2.0	1.0	5.8	7.5	5.8	0.01	0.01	6.0	0.5	2.5	0.22
Modification V	Actual	0.041	5.82	1.97	0.93	5.6	7.26	5.93	0.052	0.014	6.79	0.59	1.78	0.18



(a) TRW-NASA VIA
Modification IV



(b) TRW-NASA VIA
Modification V

Figure 25. Light photomicrographs of Task II (casting superalloy) Series III alloys showing typical as-cast microstructure from center of cast block. Magnification, 100X.

(2) Mechanical Property Characterization

(a) Tensile Test Results

Tensile tests were conducted at 927°C (1700°F) on five specimens from each of the Series III casting alloys. The results of these tests are listed in Table XIX. The evaluations included tests on specimens machined from the corners and centers of the cast block. Specimens were tested in the as-cast condition and after receiving a 899°C (1650°F) 24 hour age. This heat treatment has been applied to heavily alloyed nickel-base superalloy casting compositions to provide grain boundary carbide stabilization (53). The tensile results indicated comparable strength levels for both of the Series III alloys. As-cast specimens from the centers of the cast blocks exhibited ultimate tensile strengths in excess of 648.1 MPa (94 ksi) and yield strengths in excess of 599.8 MPa (87 ksi), surpassing the program goal of 517 MPa (75 ksi). The 24 hour aging treatment at 899°C (1650°F) did not appear to have any significant effect upon the utlimate tensile strength values, but it did have a significant effect upon the yield strength and the tensile ductility. For the Modification IV alloy the yield strength was reduced by approximately 15% by the aging treatment while Modification V alloy suffered a 10% reduction as a result of the heat treatment. In spite of the yield strength reduction, the 541.3 MPa (78.5 ksi) average value for the Modification V specimens still exceeded the program goal of 517 MPa (75 ksi). The tensile ductility, on the other hand, was significantly improved for both alloys by the aging treatment. In some instances percent elongation and reduction of area improved by as much as a factor of two. There was little evidence of the effect of specimen location on the tensile strength properties. Specimens from the corner and center locations tested in the aged condition exhibited comparable ultimate tensile strengths and yield strengths. The ductility values, in general, were lower for specimens machined from the centers of the cast blocks.

Comparison of the Series III tensile results with those for the Series I TRW-NASA VIA alloy and the three Series II modifications indicated a general improvement in tensile strength properties as a result of the Series III alloy changes. The Modification IV and V alloys exhibited the highest ultimate tensile and yield strength values of the cast superalloys. A plot of the yield strength for the Series III casting superalloys compared to the Series I TRW-NASA VIA alloy and the Modification II composition (the best of the Series II alloys) is shown in Figure 26. All of the yield strength data values were included in this plot for each of the alloys. The superiority in yield strength of the Series III alloys compared to the other two compositions is evident.

(b) Creep-Rupture Test Results

Creep-rupture tests were conducted at 954°C (1750°F)/206.8 MPa (30 ksi) on nine specimens from each of the Series III alloys and the results of these tests are listed in Table XX. For each of the alloys specimens were machined from both the corner and center locations. Tests were conducted on material in both the as-cast condition and after a 24 hour age at 899°C (1650°F). Analysis of the creep-rupture results overall indicated that Modification IV exhibited better rupture life than the Modification V alloy, but was slightly inferior in terms of time to reach 0.1% creep. Comparing average rupture lives for aged specimens machined from the corners of the cast blocks, for example, indicated that Modification IV exhibited 157.9 hours average life while Modification V exhibited 139.7 hours. For aged specimens from the center portions of the

Table XIX

Task II (Casting Superalloys) Series III 927°C (1700°F) Tensile Test Results

Alloy	Location	Heat Treatment	Ultimate Tensile Strength MPa	Ultimate Tensile Strength Ksi	0.2% Offset Yield Strength MPa	0.2% Offset Yield Strength Ksi	% Elongation	% Reduction Area
TRW-NASA VIA	(2)	As Cast	655.7	95.1	613.6	89.0	2.5	0.8
Modification IV	(1)	Aged (3)	656.4	95.2	492.3	71.4	5.5	6.6
	(2)	"	639.2	92.7	517.8	75.1	1.9	1.6
	(2)	"	666.8	96.7	537.2	77.9	2.4	0.4
TRW-NASA VIA	(2)	As Cast	650.2	94.3	603.7	87.7	2.2	2.0
Modification V	(1)	Aged (3)	659.9	95.7	512.3	74.3	4.7	6.6
	(2)	"	627.4	91.0	546.8	79.3	4.1	5.5
	(1)	"	668.8	97.0	541.3	78.5	5.6	7.4
	(2)	"	667.7	96.8	565.4	82.0	2.4	3.5

Air Force Program Goal - 517 MPa (75 Ksi) 0.2% offset yield strength at 927°C (1700°F) in a section size of 2.8 cm (7 inches)

(1) Machined from corner of cast block
(2) Machined from center of cast block
(3) Age: 899°C (1650°F)/24 hrs air cool

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EXPLORATORY DEVELOPMENT OF DIE MATERIALS FOR ISOTHERMAL FORGING-ETC(U)
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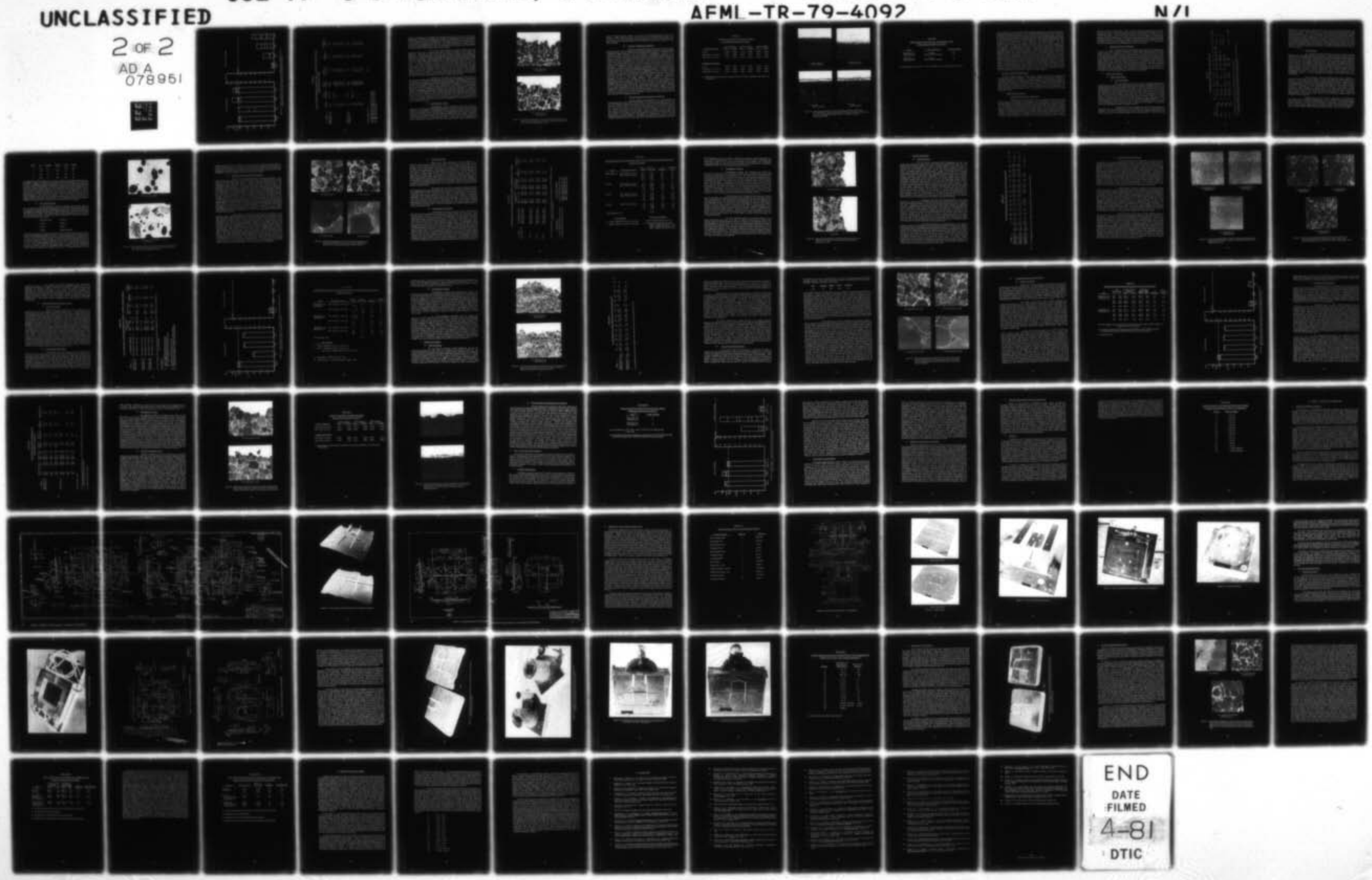

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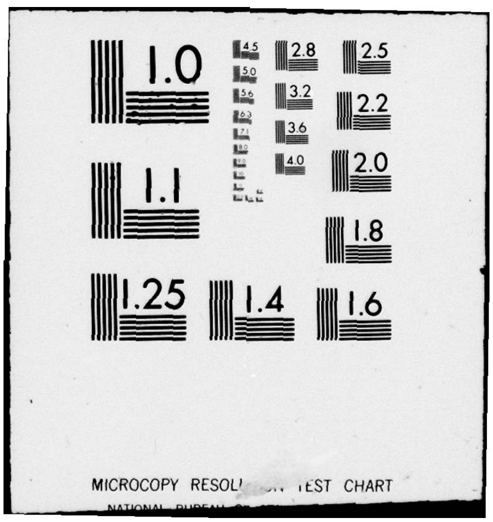
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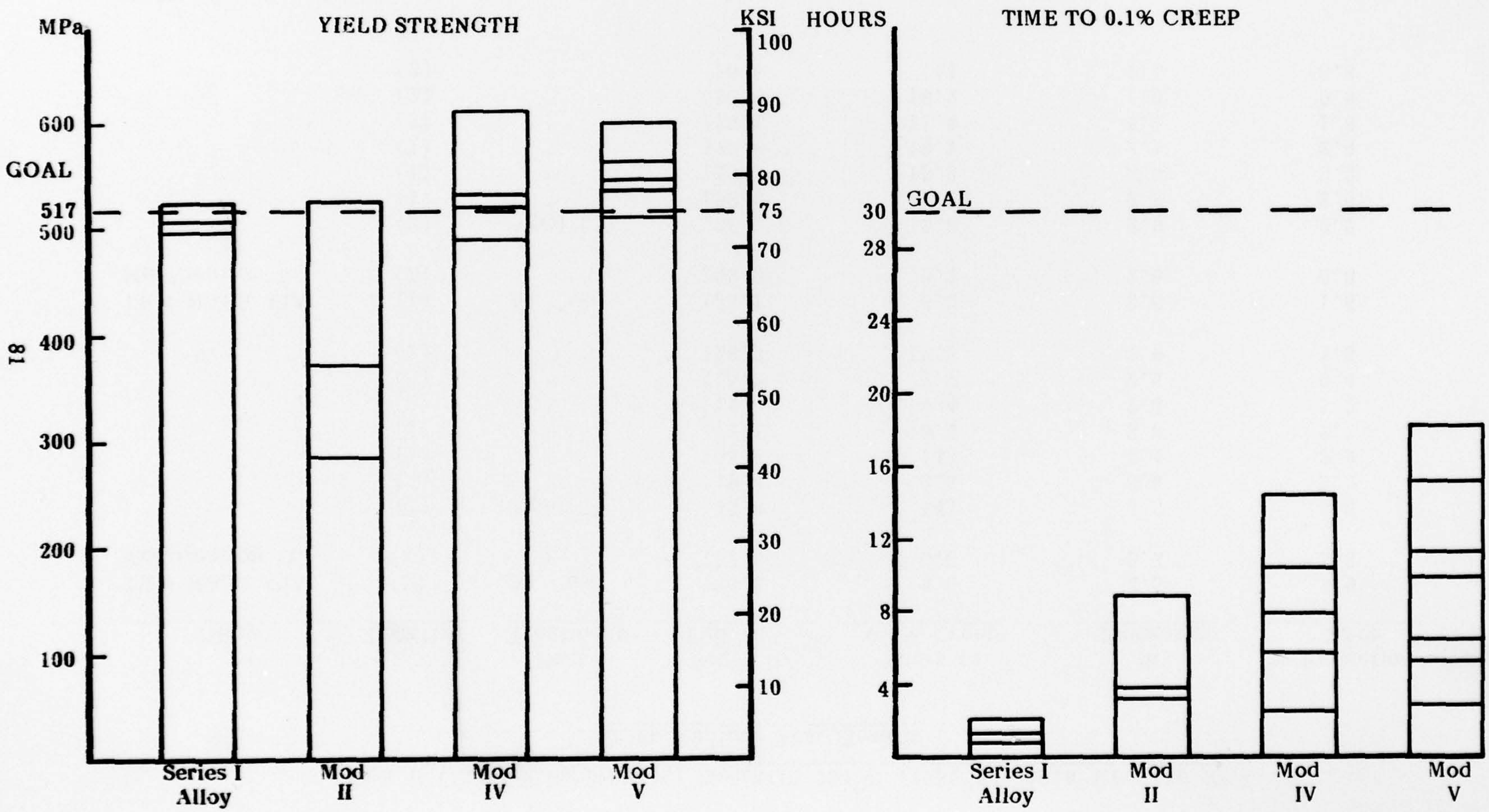


Figure 26. Yield strength and time to 0.1% creep for the Task II (casting superalloy) Series III compared to Series I TRW-NASA VIA and Series II Modification II alloys.

Table XX

Task II (Casting Superalloys) Series III 954°C (1750°F)/206.8 MPa (30 Ksi)

Creep Rupture Test Results

Alloy	Location	Heat Treatment	Hours to Failure	Hours to 0.1% Creep	% Elongation	% Reduction Area
TRW-NASA VIA Modification IV	(1)	As Cast	156.3	8.0	3.5	3.9
	(2)	"	112.6	5.8	2.3	3.8
	(1)	Aged(3)	142.8	(4)	3.2	3.5
	(1)	"	149.4	2.6	3.6	3.9
	(1)	"	168.6	(4)	4.4	3.9
	(1)	"	171.1	10.4	3.6	2.9
	(2)	"	114.0	14.4	2.8	1.2
	(2)	"	115.9	2.7	2.5	0.4
	(2)	"	128.3	10.5	2.9	1.6
TRW-NASA VIA Modification V	(1)	As Cast	135.1	6.6	2.6	1.6
	(2)	"	109.2	3.2	2.6	0.8
	(2)	Aged (3)	136.3	5.6	2.9	2.7
	(1)	"	138.9	5.3	2.8	3.5
	(1)	"	159.3	15.3	3.0	3.2
	(1)	"	120.9	10.1	2.1	2.4
	(2)	"	128.1	11.4	2.7	1.6
	(2)	"	80.6	18.2	1.6	0.8
	(2)	"	76.6	(4)	1.5	0.8
AFML Program Goal				30.0		

(1) Machined from corner of cast block
(2) Machined from center of cast block
(3) Age: 899°C (1650°F)/24 hrs air cool
(4) Recorder malfunction

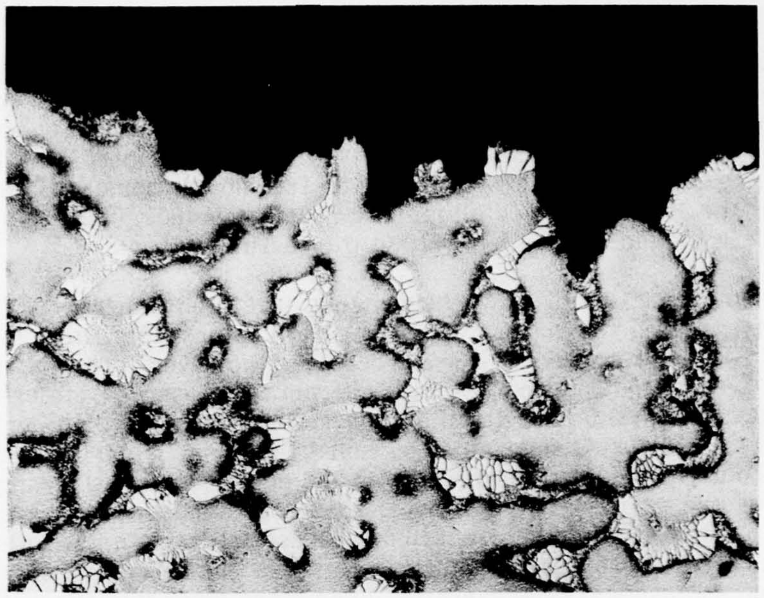
castings Modification IV exhibited an average rupture life of 119.4 hours while Modification V exhibited an average of 105.4 hours life. Modification IV was also superior to Modification V in the as-cast condition for similar specimen locations. In terms of time to reach 0.1% creep, however, Modification V was slightly superior to the Modification IV alloy, exhibiting a range of times from 5.3 to 18.2 hours, compared to a range of 2.6 to 14.4 hours for the Modification IV alloy. The ductility values, in general, were comparable for specimens from similar locations within the castings.

The 24 hour aging treatment at 899°C (1650°F) had little significant effect upon the rupture lives and the rupture ductilities for the various test specimens. The time to reach 0.1% creep was, however, effected by the aging heat treatment. As-cast Modification IV material from the corner of the casting, for example, reached 0.1% creep in 8.0 hours, while aged material from the corner exhibited a range from 2.6 to 14.4 hours. As-cast Modification V material from the corner reached 0.1% creep in 6.6 hours, while aged material from the corner exhibited a range from 5.3 to 15.3 hours. It must be pointed out, however, that the as-cast values represent single test points. The effect of specimen location was consistent in these alloys and was more pronounced in terms of rupture life than in terms of time to reach 0.1% creep. For both alloys, and for as-cast and aged material, specimens from the corner locations exhibited superior rupture lives compared to the specimens from the centers of the castings. Neither the times to reach 0.1% creep nor rupture ductility appeared to be effected by the specimen location. In fact, for both alloys, the longest times to reach 0.1% creep were exhibited by specimens machined from the center portion of the castings.

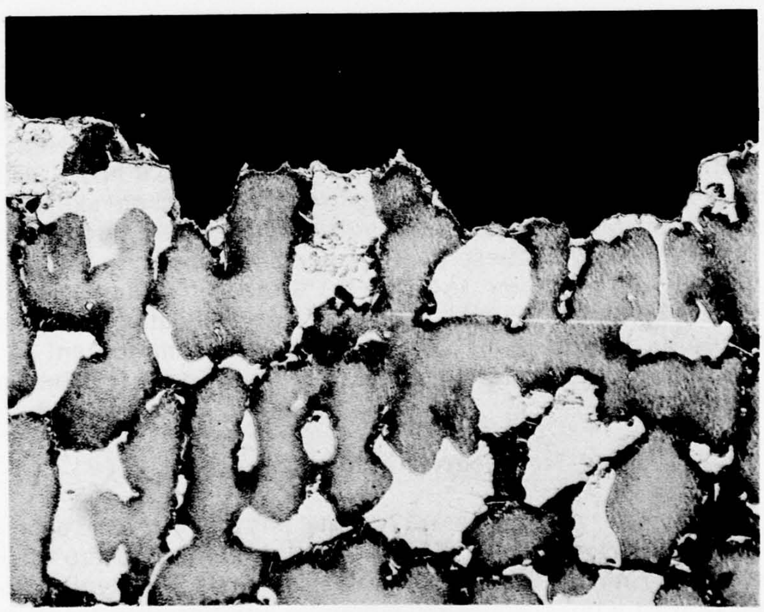
Comparison of the Series III creep rupture results with those for the Series I TRW-NASA VIA alloy and the three Series II modifications indicated an improvement as a result of the Series III chemistry changes. The Modification IV and V alloys exhibited considerable improvement in rupture life compared to the Series II alloys, but were still somewhat inferior to the original Series I composition For example, the Series I alloy exhibited a maximum rupture life value of 187.6 hours, while the best Series III rupture life was recorded by a Modification IV specimen exhibiting 171.0 hours to failure. Considerable improvement, however, was observed in the times to reach 0.1% creep for the Series III alloys. These improvements are shown in Figure 26, which includes a plot of all the test values of time to reach 0.1% creep for the Series III alloys compared to the Series I base composition and the Modification II alloy (the best of the Series II alloys). These results indicated that the increased hafnium content in combination with the rhenium addition of the Series III alloys was responsible for the improvement in the times to reach 0.1% creep.

(c) Metallographic Analysis

Metallographic analysis conducted upon failed test bar specimens of the two Series III alloys indicated that, similar to the other cast superalloys evaluated in this program, an intergranular failure mode was typical for both the tensile and the creep rupture tests. Typical examples of intergranular fracture paths are shown in Figure 27 for creep rupture test specimens of Modification IV and V alloys. For both of these alloys the fracture paths were associated with the large, eutectic colonies of primary gamma-prime. The fracture path shown in Figure 27b for the Modification V alloy is particularly interesting in that the entire path appears almost continuous with



(a) TRW-NASA VIA
Modification IV



(b) TRW-NASA VIA
Modification V

Figure 27. Light photomicrographs of Task II (casting superalloys) Series III alloys showing intergranular failure mode typical of creep-rupture test specimens. Magnification, 100X.

either the large eutectic colonies or with the interconnecting regions between the colonies. While continuous paths such as these have previously been shown to be detrimental to the high temperature mechanical properties of nickel-base superalloys (54), their formation was clearly beneficial to the tensile and creep-rupture properties of the Series III alloys.

(d) Lubricant Compatibility Evaluation

Lubricant compatibility tests were conducted on the Series III alloys using OPT 112 and Deltaglaze 69 isothermal forging lubricants. Testing consisted of 80 hours total exposure at each of three temperatures including 871°C (1600°F), 927°C (1700°F) and 982°C (1800°F). The tests included a metallographic evaluation of the oxidation/sulfidation attack and involved measurements of the depth of penetration of the attack. As a direct comparison with the currently used superalloy isothermal forging die material, tests were also included on IN-100 material from the Series I study. The results of these lubricant compatibility tests are shown in Table XXI in terms of the depth of attack. The results indicated that for the Series III alloys the Deltaglaze 69 lubricant was somewhat more aggressive than the OPT 112 in its attack on the modified TRW-NASA VIA superalloys. It exhibited a greater tendency for spalling as well as a subsurface attack. The mode of attack for both of the forging lubricants, however, involved a general surface corrosion rather than a localized penetration along grain boundaries. Although the depth of penetration was somewhat greater for the Modification V alloy in terms of actual numbers, there appeared to be little significant difference between the two alloys in their resistance to attack. This reflected their overall similarity in compositions. Comparison with the data for the IN-100 alloy, however, indicated a significant difference in resistance to attack by the forging lubricants. The resistance to attack of the Modification IV and V alloys was superior to that of IN-100 at all three temperatures. Another difference was observed in that the OPT 112 lubricant was more aggressive than Deltaglaze 69 in its attack upon IN-100. The mode of attack on the IN-100 was similar to that of the Series III alloys and involved a generalized surface type corrosion. Examples of the typical appearance of this corrosion attack are shown in Figure 28 for the Modification V alloy and the IN-100 after 80 hours exposure at 927°C (1700°F) and 982°C (1800°F). In summary, the lubricant compatibility results demonstrated that the Series III alloy modifications exhibited superior resistance to attack by forging lubricants than the currently used IN-100 forging die material.

(e) Thermal Fatigue/Creep Interaction Test Results

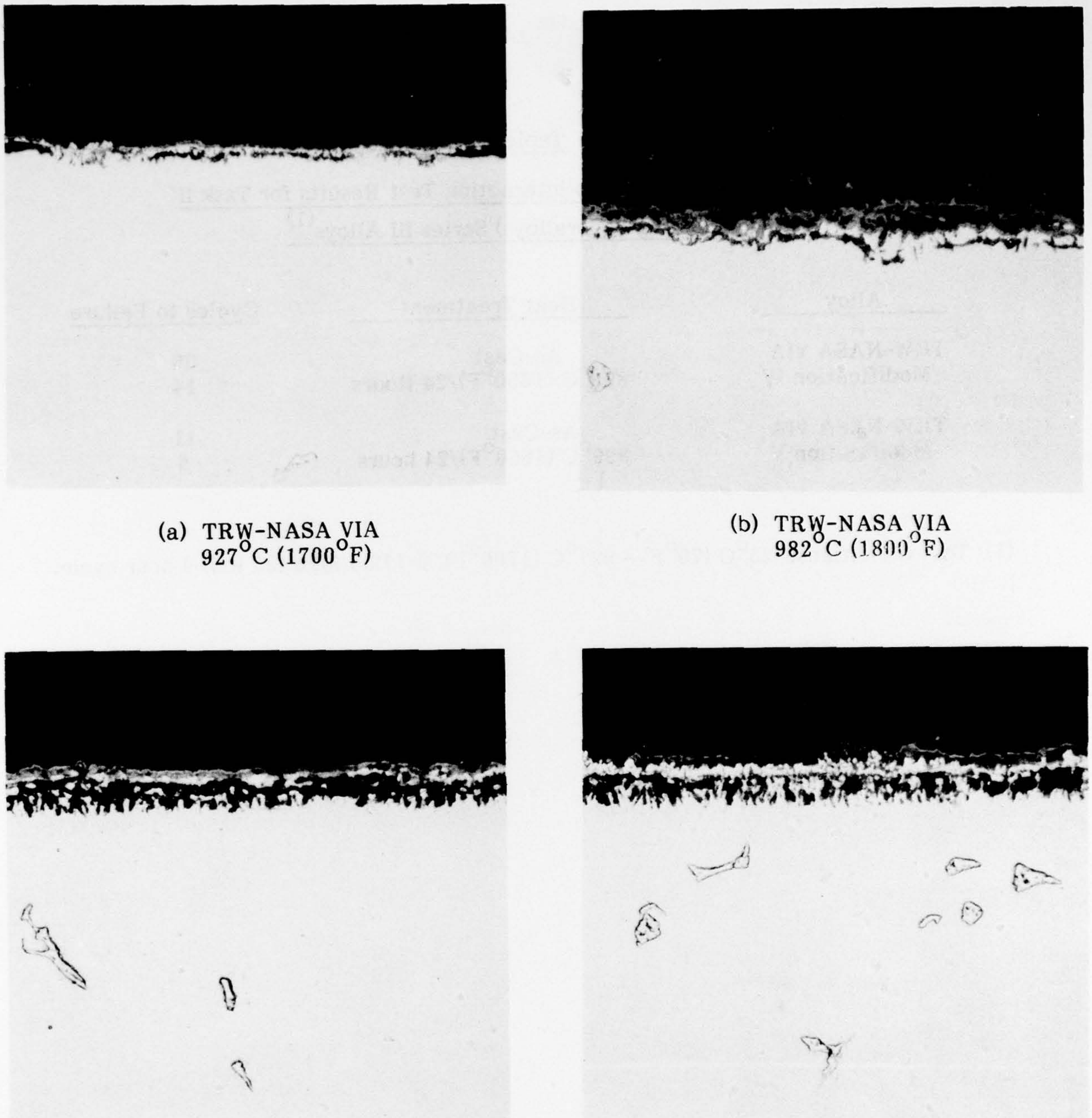
Thermal fatigue/creep interaction tests were conducted on two specimens from each of the Series III alloys. Test specimens were machined from the center portions of the cast blocks and were tested in the as-cast condition and after a 24 hour age at 899°C (1650°F). The specimens were loaded at 927°C (1700°F)/172 MPa (25 ksi). The test cycle ran for four hours under load and temperature after which specimens were unloaded and cooled at the same time to room temperature for 5-10 minutes and then reheated and loaded for the next cycle. Cycles to complete specimen separation were measured. The results of these thermal fatigue tests are listed in Table XXII. The data indicate that Modification IV was superior to Modification V alloy in thermal fatigue/creep interaction resistance. In addition, material which was tested in the as-cast condition exhibited lives two to two and a half times greater than material in the heat-

Table XXI

Lubricant Compatibility Test Results for Task II (Casting Superalloy) Series III Alloys(1)

OPT 112 Lubricant	871°C (1600°F) mm	871°C (1600°F) inch	927°C (1700°F) mm	927°C (1700°F) inch	982°C (1800°F) mm	982°C (1800°F) inch
IN-100	.00965	.00038	.01270	.00050	.02235	.00088
TRW-NASA VIA Mod IV	.00127	.00005	.00635	.00025	.00737	.00029
TRW-NASA VIA Mod V	.00152	.00006	.00762	.00030	.00965	.00038
Deltaglaze 69 Lubricant						
IN-100	.00330	.00013	.01270	.00050	.01905	.00075
TRW-NASA VIA Mod IV	.00203	.00008	.00864	.00034	.01092	.00043
TRW-NASA VIA Mod V	.00229	.00009	.00457	.00018	.01321	.00052

(1) Measurements indicate depth of penetration after 80 hours exposure at the various test temperatures.



(a) TRW-NASA VIA
927°C (1700°F)

(b) TRW-NASA VIA
982°C (1800°F)

(c) IN-100
927°C (1700°F)

(d) IN-100
982°C (1800°F)

Figure 28. Light photomicrographs of Task II (casting superalloys) TRW-NASA VIA Modification V alloy and IN-100 after 80 hours lubricant compatability test with Deltaglaze 69 forging lubricant. Unetched condition. Magnification, 500X.

Table XXII

Thermal Fatigue/Creep Interaction Test Results for Task II (Casting Superalloys) Series III Alloys[1]

Alloy	Heat Treatment	Cycles to Failure
TRW-NASA VIA Modification IV	As-Cast	36
	899°C (1650°F)/24 Hours	14
TRW-NASA VIA Modification V	As-Cast	11
	899°C (1650°F)/24 hours	6

(1) Test Conditions: 25°C (70°F) - 927°C (1700°F), 0-172.4 MPa (25 Ksi) 4 hour cycle.

treated condition. Since the 899°C (1650°F) aging temperature lies within the anticipated temperature range of application for the isothermal forging dies, these results implied a deterioration in fatigue resistance of the die materials with prolonged usage. Analysis of the fatigue results in relation to the tensile and creep-rupture results presented previously for the Series III alloys offered several observations. These observations applied to both of the Series III compositions. First, fatigue life was directly related to the yield strength, and second, it was inversely related to the tensile ductility. This was determined from a comparison between heat-treated and as-cast tensile properties at 927°C (1700°F). The aging heat treatment had the effect of reducing the yield strength and fatigue life, while increasing the ductility. The inverse ductility effect was difficult to rationalize in terms of previous studies on creep-fatigue interactions in nickel-base superalloys tested under isothermal conditions. These studies indicated that fatigue lives generally increase with tensile ductility because of the enhanced ability of the more ductile material to accomodate creep strain deformation (55). Apparently this type of behavior was not characteristic of the Series III alloys tested under the more complex loading conditions of the present study in which temperature (and hence ductility at the particular temperature) was changing during the heating and cooling portions of the load cycle. It was also observed that no relationships were apparent between the fatigue properties and the ultimate tensile strength and the creep-rupture properties of these alloys. Metallographic analysis was conducted on failed fatigue test specimens and indicated that an intergranular failure mode, similar to that for the tensile and creep rupture test specimens, was common to both alloys. There was little difference between the various specimens to explain the variations in fatigue response.

C. Task III - Metastable Carbide Alloys

The Task III work included evaluation of nine candidate alloy compositions produced by metastable carbide techniques. Metastable carbide processing was designed to overcome the problem of the presence of stable carbides in the grain boundary regions of superalloy powder compacts which prevented the important grain growth necessary for improved high temperature stress rupture properties. The nine alloys were divided into three series with four alloys in Series I, three in Series II and two in Series III. The following sections contain descriptions of the procedures used for the development of these metastable carbide alloys as well as a discussion of the results of the alloy development effort.

1. Experimental Procedures

a. Powder Preparation and Consolidation

Hydrogen gas atomization was used to produce the carbon-free master powder alloys for this portion of the program. In this procedure, 22.6 Kg (50 pound) vacuum induction melted ingots of the desired master alloy composition were produced at the TRW Turbine Components Metals Division and then shipped to Homogeneous Metals, Inc., for atomization. Procedures identical to those used for the Task I powder metallurgy alloys were used for the atomization of the master alloys. Attrition was then utilized to produce a homogeneous distribution of metastable carbide particles throughout the powder mixture. To accomplish this the proper amounts of metastable carbide additions were attrited with the prealloyed base powder in the TRW 10-S Szegvari Attritor, under a protective argon atmosphere, to produce the desired alloy composition with a minimum

amount of impurity contamination. Close in-house control was maintained of the entire attrition operation to insure that complete particle comminution was obtained for these experimental alloys. Various attrition parameters were studied for each alloy and optimum parameter selection was made on the basis of the degree of homogeneity achieved in the attrited mixture. The powders were compacted by hot isostatic pressing at Industrial Materials Technology utilizing can configurations and procedures described previously for the powder metallurgy alloys. Similar to this Task I work a parametric study was conducted to insure complete densification of the powders.

b. Mechanical Property Evaluations

The mechanical property evaluations were similar to those conducted on the Task I and II alloys. Mechanical property screening studies including tensile and creep rupture tests were conducted on the Series I and II alloys while the more extensive mechanical property characterizations were conducted on the two Series III alloys. Prior to the mechanical property screening evaluations, a heat treatment study was conducted to develop the optimum grain structure with a discrete particle carbide morphology in the grain boundaries for each metastable carbide alloy. In general, the heat treatment involved three steps, including a high temperature grain coarsening anneal, a partial solutioning treatment and a low temperature aging treatment. The grain coarsening anneal solutioning was conducted to accomplish two objectives: (1) to solution the metastable carbide thus freeing carbon for subsequent precipitation, and (2) to develop the desired grain size and morphology. The procedures for the mechanical property tests were all similar to those described previously for the Task I and II alloys.

2. Results and Discussion

(1) Series I Alloy Studies

(a) Alloy Formulation

The four alloys selected for the Series I metastable carbide alloy development work were similar to those for the Task I powder metallurgy and the Task II casting superalloy efforts and included TRW-NASA VIA, TAZ-8A, IN-792, and IN-100. The specific alloy compositions selected for the Series I work were identical to the powder metallurgy alloys. An important difference between the two types of alloys, however, was that the carbon in the metastable carbide alloys was added in the form of SiC to carbon free base alloy powders during attrition processing. The aim compositions of the four Series I alloys are listed in Table XXIII along with the actual compositions in weight percent of material processed during the HIP parametric study. Specimens of this particular material were used for the mechanical property screening evaluations. For all of the metastable carbide alloys the carbon content aim was reduced from the normal levels characteristic of the casting versions to enhance the development of large grain sizes in the consolidated powder metallurgy material.

Comparison of the aim compositions with the actual chemistries indicated that, for the IN-792 and the IN-100 alloys, the desired compositions were achieved. For both the TRW-NASA VIA and the TAZ-8A compositions, however, the carbon contents were considerably higher than the desired levels. In fact, the actual

Table XXIII

Task III (Metastable Carbide Alloy) Series I Alloy Compositions (1)

Alloy		C(2)	C	Cr	Mo	Ti	Al	Co	W	Zr	B	Ta	Cb	Hf	Si	N_2	O_2	Other
TRW-NASA VIA	Aim	0.13	0.05	6.1	2.0	1.0	5.4	7.5	5.8	0.13	0.02	9.0	0.5	0.43				
	Actual		0.13	5.63	2.01	0.88	5.29	7.73	6.01	0.059	0.015	9.38	0.65	0.51	0.36	310	46	
TAZ-8A	Aim	0.13	0.05	6.0	4.0		6.0		4.0	1.0	0.004	8.0	2.5					
	Actual		0.14	6.17	3.9		5.87		4.35	1.05	0.012	8.12	2.53		0.29	340	42	
IN-792	Aim	0.12	0.05	12.4	1.9	4.5	3.1	9.0	3.8	0.10	0.02	3.9			0.18	290	51	
	Actual		0.03	12.25	1.88	4.15	3.05	9.02	3.39	0.053	0.028	3.65						
IN-100	Aim	0.18	0.05	10.0	3.0	4.7	5.5	15.0		0.06	0.016							1.0V
	Actual		0.04	8.83	2.99	4.5	5.97	14.5		0.067	0.016				0.15	285	29	0.73V

(1) In weight percent, except for oxygen and nitrogen which are reported in parts per million.

(2) These are the nominal carbon contents for these alloys as produced in the cast form. Carbon has been lowered for the metastable carbide study to develop large grain size (ASTM 2-3) in the consolidated powder.

carbon levels for these alloys approximated those for the casting alloy compositions. The high carbon contents resulted from an error made involving the addition of SiC during attrition processing. The two compositions were included in the mechanical property screening studies because, in spite of the high carbon levels, HIP processing did result in large grain sizes in the microstructures. These results will be discussed in greater detail in the section describing the HIP parametric study. Of potentially greater concern were the relatively high oxygen levels of the metastable carbide alloys. In spite of the fact that attrition operations were conducted under a protective argon atmosphere, the oxygen levels were considerably higher than those for the powder metallurgy alloys, Tables I, IV and VII. These high oxygen levels reflected the increased handling involved with the attrition process.

(2) Attrition Study

The attrition study was conducted on the Series I alloys in order to determine the processing parameters required to achieve proper mixing of the SiC particles with the carbon-free base powders with a minimum amount of contamination. Attrition of the mechanically alloyed oxide dispersion strengthened (ODS) materials usually requires times of approximately 40 hours to achieve a proper oxide dispersion in those alloys (56). The purpose of using attrition in the present investigation was not so much to achieve a fine dispersion of SiC particles throughout the structure but rather to obtain enough mixing so that subsequent HIP processing would result in a uniform, large grained structure. Previous efforts concerning metastable carbide processing employed simple tumbling methods to achieve mixing (8). The resultant blends were not homogeneous in terms of either the carbide distribitons or the grain sizes of the consolidated powders. It was anticipated that attrition could improve upon these distributions.

The preliminary attrition processing trials were conducted on carbon-free TRW-NASA VIA base powder to which were added the proper amounts of 7 micron or less SiC particles to achieve a carbon content of 0.05%. Previous studies utilizing mild carbon steel balls for the attrition process have resulted in typical iron pick-up of approximately 1-2% in similar nickel-base superalloys (57). Some success has been achieved in minimizing this iron contamination by running a wash heat of similar composition powder through the attritor prior to the actual experimental attrition operation (58). This results in a protective covering of a nickel-base alloy on the carbon steel balls and attritor arms which reduces iron contamination. Such a wash heat (approximately 4 hours attrition time) was run in the present investigation which did minimize iron contamination.

Initially, three attrition runs were conducted on the TRW-NASA VIA powders, using a ball/powder charge ratio of 16/1. Attrition was conducted under an argon atmosphere to minimize contamination of the powders. These trials included attrition times of 1, 2, and 4 hours. The results of these trials are indicated below in terms of iron, nitrogen, oxygen, carbon and silicon contents. Iron, carbon and silicon are given in weight percent, while nitrogen and oxygen are given in parts per million (ppm).

Hours	Iron	Nitrogen	Oxygen	Carbon	Silicon
0	.040	29	170	.002	.065
1	.055	46	310	.05	.23
2	.066	42	310	064	.19
4	.078	51	340	.062	.18

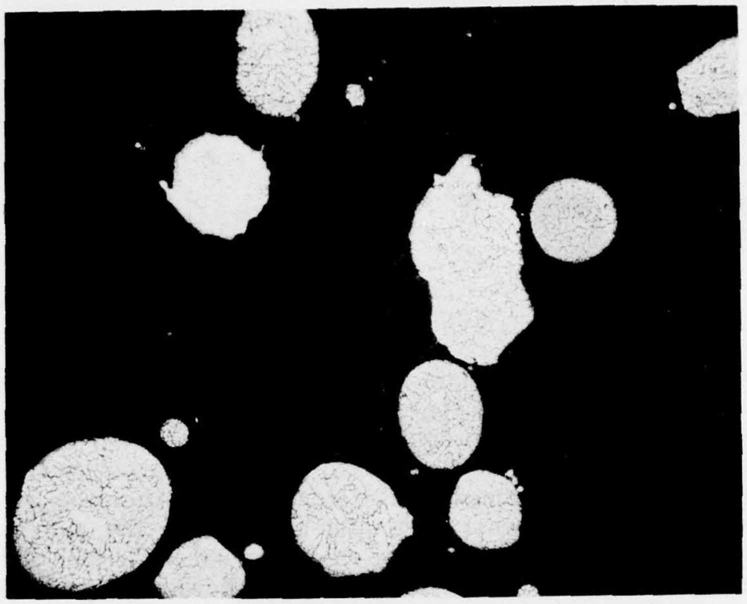
It was observed that an attrition time of 4 hours resulted in the desired carbon content in the powders. Oxygen and nitrogen contamination occurred after the first hour of attrition and while a significant increase in contamination level was not observed with increasing attrition time, the trends appeared to indicate that longer attrition times would result in a prohibitive increase in these elements. The iron contamination was not excessive in terms of the normal 1-2% pick-up reported in the literature. The increased silicon content reflected the fact that SiC particles were used to make the carbon additions. The typical microstructure of particles attrited for 4 hours is shown in Figure 29, which includes a photomicrograph of the as-received powders. While it was evident that some of the powders (approximately 25%) were not completely worked and retained some of their as-atomized dendritic structure, considerations of the oxygen and nitrogen contamination resulted in the selection of an attrition time of 4 hours for the Series I alloys.

(3) HIP Parametric Study

The HIP parametric study was conducted on the Series I alloys in order to determine the processing conditions required to achieve complete densification in the consolidated powders and the desired ASTM 2-3 grain size. The initial HIP condition investigated for each alloy included 1316^{o}C (2400^{o}F)/103.4 MPa (15 ksi) for four hours and was selected on the basis of the success achieved with these parameters in the Task I powder metallurgy efforts. The grain size data resulting from this processing condition are given below for the four alloys:

TRW-NASA VIA	ASTM 2-4
TAZ-8A	ASTM 2-3
IN-792	ASTM 2
IN-100	Specimen Melted

These grain size results compared quite favorably with the values exhibited by the Series I powder metallurgy alloys, Figures 4, 5 and 6. It was apparent that the higher carbon contents of the TRW-NASA VIA and TAZ-8A alloys did not prevent the achievement of large grain sizes in the HIP'ed material. In fact, the metastable carbide TAZ-8A alloy was able to be HIP'ed at a higher temperature and resulted in a larger grain size than in the Task I powder metallurgy alloy (ASTM 2-3 versus 3-5). Since the IN-100 specimen melted at the 1316^{o}C (2400^{o}F) HIP temperature, an alternative HIP consolidation operation at 1288^{o}C (2350^{o}F) was investigated which resulted in complete densification without incipient melting and an ASTM 3-4 grain size. Although this grain size was



(a) As received powder

(b) Powder after 4 hours attrition

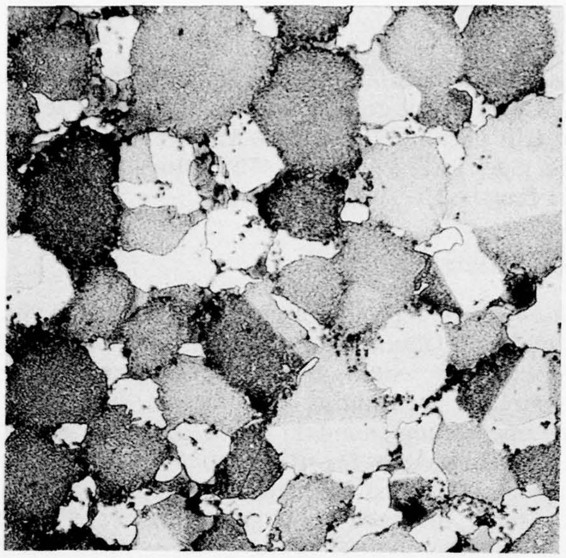
Figure 29. Light photomicrographs of Task III (metastable carbide) Series I TRW-NASA VIA alloy showing effects of 4 hours attrition on the microstructure. Magnification, 100X.

somewhat smaller than that exhibited by the Task I powder metallurgy IN-100 HIP'ed at 1288°C (2350°F), Figure 7c, it was considered relatively comparable to the other Series I metastable carbide alloys. As a result of the HIP parametric study a 1316°C (2400°F) HIP temperature was selected for the TRW-NASA VIA, TAZ-8A and IN-792 alloys while a HIP temperature of 1288°C (2350°F) was selected for the IN-100 alloy.

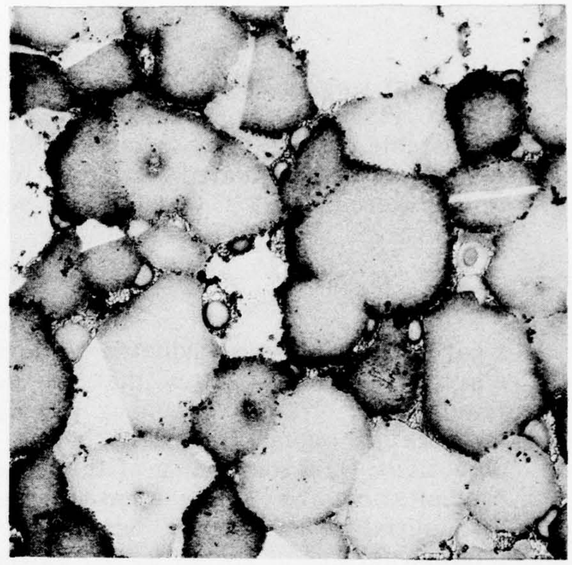
(4) Mechanical Property Screening Evaluation

Prior to the mechanical property screening evaluations a heat treatment study was conducted to determine the optimum test microstructure for each alloy. It was anticipated that the heat treatments would involve three steps, a high temperature grain coarsening anneal, a partial solutioning treatment and a low temperature aging treatment. The grain coarsening anneal would accomplish two objectives: (1) a solutioning of the metastable carbide thus freeing carbon for subsequent precipitation, and (2) an increase in grain size compared to the HIP consolidated powders. The initial phase of the heat treatment study involved a characterization of the microstructures of the as-HIP'ed alloys. This analysis indicated the presence of carbides both in the grain boundaries and within the grains themselves. Microprobe analysis, however, revealed that these were not the anticipated metastable SiC particles. The carbides were, in fact, rich in titanium or tantalum depending upon the presence of these elements in the particular alloy compositions. The relative stability of these carbides was demonstrated by the fact that they could not be solutioned at temperatures approaching 1288°C (2350°F) - 1316°C (2400°F). Consequently, little additional grain growth occurred within the microstructures. These results suggested that during HIP consolidation, decomposition of the relatively unstable SiC particles occurred, and the carbon immediately recombined with titanium and/or tantalum to form more stable carbides. These carbides could not be solutioned and retarded further grain growth during the heat treatment of these alloys.

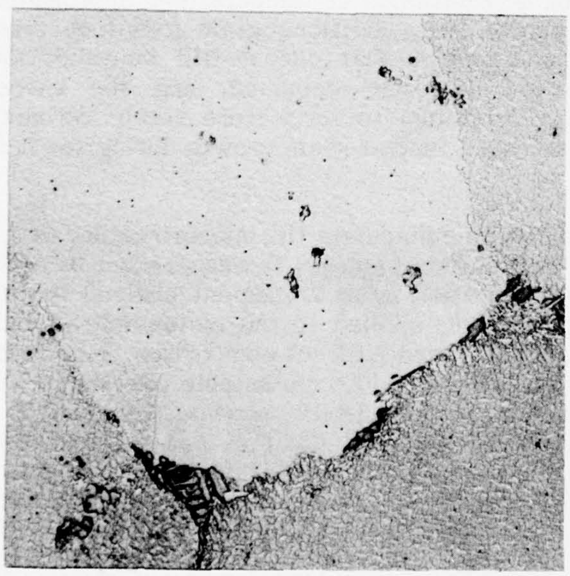
As a result of this inability to manipulate the microstructure to any significant degree through alteration of the carbide morphology, it was decided to apply the same 24 hour 899°C (1650°F) carbide stabilization aging treatment utilized for the Task I powder metallurgy alloys. This treatment was applied to the metastable carbide TRW-NASA VIA, TAZ-8A, and IN-792 alloys and allowed a direct comparison to be made between the two powder processing approaches. Because the metastable carbide IN-100 exhibited a grain size comparable to the finer grain sized IN-100 processed in Task I, it was given the same standard solution and age treatment including 1204°C (2200°F)/4 hours air cool + 1080°C (1975°F)/4 hours/air cool + 871°C (1600°F)/16 hours/air cool + 760°C (1400°F)/ 24 hours/air cool. Typical microstructures for the metastable carbide alloys in the HIP'ed and aged condition are shown in Figure 30 for the TAZ-8A and IN-792 alloys. The high magnification photomicrographs of Figure 30c and d indicate the presence of carbide particles both within the grains and along the grain boundaries. These microstructures may be compared to those presented in Figure 5 (for TAZ-8A) and Figure 6 (for IN-792) for the Task I powder metallurgy alloys. The grain size of the metastable carbide TAZ-8A is considerably larger than that of the powder metallurgy alloy, while that for the IN-792 alloy is comparable to that of the powder metallurgy alloy.



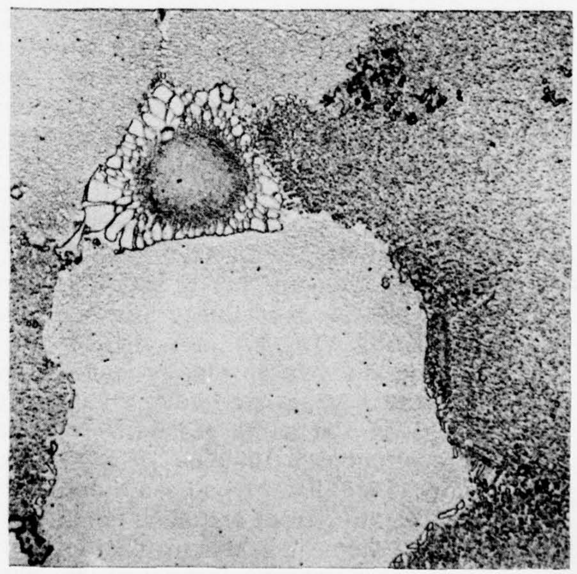
(a) TAZ-8A, 100X



(b) IN-792, 100X



(c) TAZ-8A, 500X



(d) IN-792, 500X

Figure 30. Light photomicrographs of Task III (metastable carbide) Series I TAZ-8A and IN-792 alloys after HIP at 1316°C (2400°F)/103.4 MPa (15 ksi)/4 hours plus 24 hours age at 899°C (1650°F).

(a) Tensile Test Results

Tensile tests were conducted at 927°C (1700°F) on a minimum of three specimens from each of the Series I metastable carbide alloys and the results of these tests are listed in Table XXIV. These results indicated that, on the average, both the TRW-NASA VIA and the IN-792 exhibited yield strengths meeting the 517 MPa (75 ksi) program goal. The average yield strengths for the TRW-NASA VIA and IN-792 were 517 MPa (75 ksi) and 519.9 MPa (75.4 ksi), respectively. The TRW-NASA VIA, did exhibit a slightly higher ultimate tensile strength than the IN-792, but only by approximately 17 MPa (5 ksi). In comparison to the powder metallurgy versions of these alloys, the metastable carbide TRW-NASA VIA was comparable in strength, while the IN-792 was significantly improved both in ultimate tensile strength and in yield strength. It was apparent that the high oxygen contents of the metastable carbide alloys in comparison to the powder metallurgy compositions did not adversely affect the tensile strength properties. The ductility properties were, on the other hand, reduced in comparison to the powder metallurgy alloys.

The IN-100 and the TAZ-8A metastable carbides exhibited yield strengths which were significantly lower than the TRW-NASA VIA and IN-792 alloys. The average yield strength of IN-100 was 437.2 MPa (63.4 ksi) while that for TAZ-8A was 395.8 MPa (57.4 ksi). In comparison to the powder metallurgy alloys, the IN-100 had comparable yield strength while the TAZ-8A was lower in yield strength. The ductility properties for the metastable carbide IN-100 were comparable to the powder metallurgy alloy while those for the metastable carbide TAZ-8A were higher. In summary, the tensile test results indicated that both the TRW-NASA VIA and IN-792 alloys exhibited approximately the same potential to act as an alloy base for the alloy development efforts for the powder metallurgy alloys. Both alloys exhibited yield strengths which met the 517 MPa (75 ksi) program goal.

(b) Creep-Rupture Test Results

Creep-rupture tests were conducted at 954°C (1750°F)/206.8 MPa (30 ksi) on five specimens from each of the Series I alloys and the results of these tests are listed in Table XXV. Unlike the Series I tensile results, but similar to the powder metallurgy Series I creep rupture results, the TRW-NASA VIA alloy exhibited the best overall creep rupture properties of the Series I metastable carbide alloys. The TRW-NASA VIA specimens had an average rupture life of 25.6 hours, more than double that of the TAZ-8A, the next best of the metastable carbide alloys. The metastable carbide alloy, however, was inferior in rupture life to the powder metallurgy TRW-NASA VIA alloy (average rupture life of 35 hours). With the exception of the TAZ-8A alloy, all of the metastable carbide compositions were inferior to their powder metallurgy counterparts in terms of rupture life. In the case of TAZ-8A, grain size differences may have been the deciding factor because of the significantly larger grain size of the metastable carbide alloy. In terms of time to reach 0.1% creep, however, the properties of the metastable carbide and the powder metallurgy alloys were similar in that all compositions exhibited relatively poor life compared to the 30 hour program goal. The TRW-NASA VIA alloy exhibited the longest lives to reach 0.1% creep, ranging from 0.45-2.02 hours. The lives for the other alloys were all inferior in that none of the specimens exceeded 0.32 hours (a

Table XXIV

Task III (Metastable Carbide Superalloys) Series I 927°C (1700°F) Tensile Test Results

Alloy	Processing Condition	Ultimate Tensile Strength MPa	Ultimate Tensile Strength Ksi	0.2% Offset Yield Strength MPa	0.2% Offset Yield Strength Ksi	% Elongation	% Reduction Area
TRW-NASA VIA	HIP Condition 1 Plus	657.8	95.4	497.8	72.2	2.9	1.8
	Heat Treat Condition 1	661.3	95.9	532.2	77.2	3.1	3.1
		668.2	96.9	528.9	76.7	3.3	1.8
		660.6	95.8	508.2	73.7	2.8	3.1
TAZ-8A	HIP Condition 1 Plus	487.5	70.7	390.3	56.6	6.9	7.4
	Heat Treat Condition 1	493.0	71.5	397.9	57.7	9.5	8.6
		499.2	72.4	399.9	58.0	8.9	8.6
IN-792	HIP Condition 1 Plus	613.6	89.0	492.3	71.4	3.0	2.5
	Heat Treat Condition 1	617.8	89.6	566.1	82.1	2.6	1.8
		613.0	88.9	502.0	72.8	2.8	1.8
IN-100	HIP Condition Plus	553.0	80.2	441.3	64.0	2.0	1.2
	Heat Treat Condition 2	564.1	81.8	430.5	61.7	3.1	2.1
		571.0	82.8	445.5	64.6	2.3	2.5

Air Force Program Goal: 517 MPa (75 Ksi) 0.2% offset yield strength at 927°C (1700°F) in a section size of 2.8 cm (7 inches).

HIP Conditions

1. 1316°C (2400°F)/103 MPa (15 Ksi)/4 Hrs
2. 1288°C (2350°F)/103 MPa (15 Ksi)/4 Hrs

Heat Treat Conditions

1. 899°C (1650°F)/24 Hrs/Air Cool
2. 1163°C (2125°F)/2 Hrs/Air Cool +
   1080°C (1975°F)/4 Hrs/Air Cool +
   871°C (1600°F)/24 Hrs/Air Cool +
   760°C (1400°F)/24 Hrs/Air Cool

Table XXV

Task III (Metastable Carbide Superalloys) Series I 954°C (1750°F)/206.8 MPa (30 Ksi) Creep Rupture Test Results

Alloy	Processing Condition	Hours to Failure	Hours to 0.1% Creep	% Elongation	% Reduction Area
TRW-NASA VIA	*HIP Condition 1 Plus*	26.7	2.02	2.0	1.5
	Heat Treat Condition 1	27.0	1.53	2.3	1.6
		27.0	0.45	3.6	3.1
		22.4	0.50	1.7	1.2
		25.1	1.20	2.5	2.1
TAZ-8A	HIP Condition 1 Plus	9.1	0.02	5.4	3.1
	Heat Treat Condition 1	11.9	0.02	5.5	6.2
		10.2	0.01	5.8	5.6
		9.6	0.01	4.5	5.5
		8.8	0.06	4.3	4.3
IN-792	HIP Condition 1 Plus	6.6	0.32	1.3	0.8
	Heat Treat Condition 1	6.4	0.02	1.7	0.6
		7.8	0.08	1.6	0.4
		5.9	0.07	2.0	0.6
		7.8	0.19	1.7	1.2
IN-100	Heat Treat Condition 2	4.5	0.13	2.0	1.6
		1.8	0.06	1.4	1.3
		1.4	0.01	2.0	0.6
		1.7	0.01	1.7	1.2
AFML Program Goal			30.00		

HIP Conditions

1. 1316°C (2400°F)/103 MPa (15 Ksi)/4 Hrs
2. 1288°C (2350°F)/103 MPa (15 Ksi)/4 Hrs

Heat Treat Conditions

1. 899°C (1650°F)/24 Hours/Air Cool
2. 1163°C (2125°F)/2 Hours/Air Cool +
   1080°C (1975°F)/4 Hrs/Air Cool +
   875°C (1600°F)/24 Hrs/Air Cool +
   760°C (1400°F)/24 Hrs/Air Cool

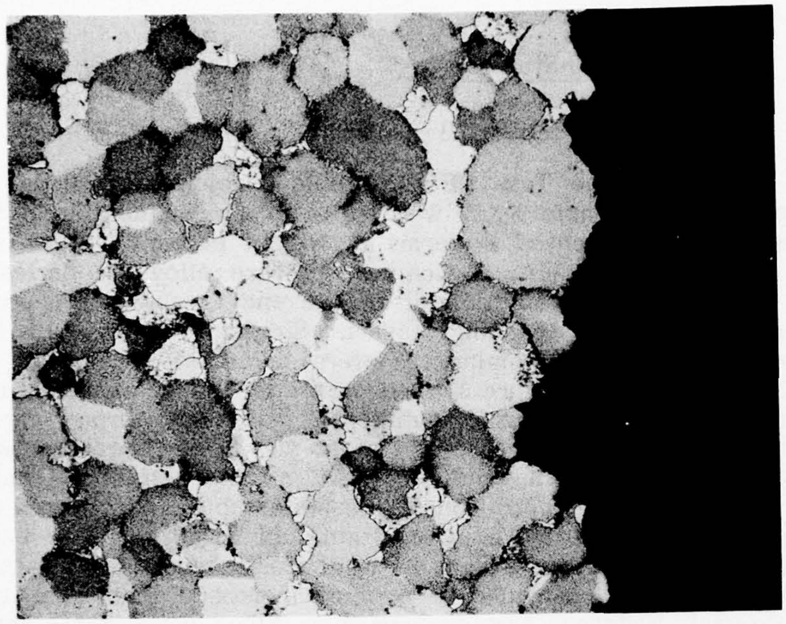
IN-792 specimen) to reach 0.1% creep. Although the metastable carbide TRW-NASA VIA alloy exhibited slightly better life to 0.1% creep than the powder metallurgy alloy (exhibiting a range of times from 0.22-1.62 hours) it was apparent that a significant improvement in time to reach 0.1% creep was required for the application of metastable carbide TRW-NASA VIA as an isothermal forging die material.

(c) Metallographic Analysis

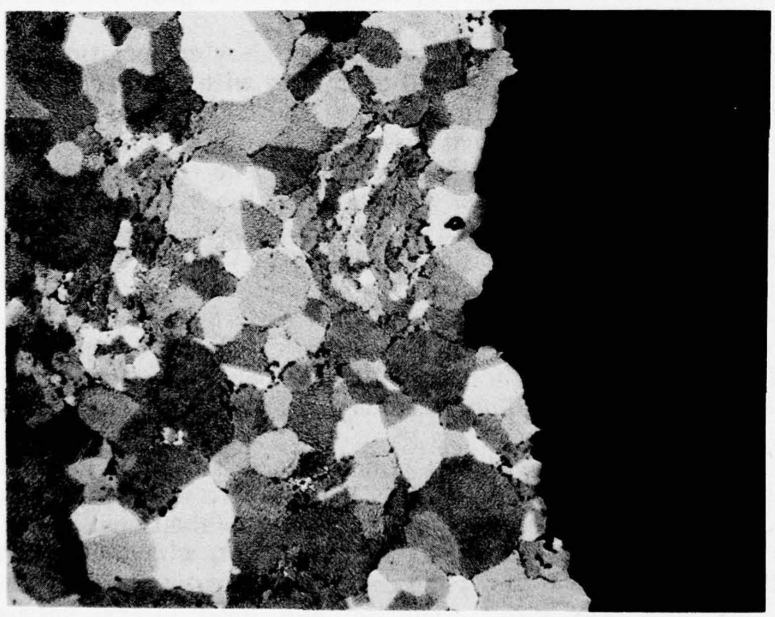
Metallographic analysis was conducted upon failed test specimens and indicated that an intergranular failure mode was typical for all four alloys for both the tensile and the creep-rupture tests. The results for the TRW-NASA VIA, TAZ-8A and IN-792 alloys were similar to those for the Task I powder metallurgy alloys in that gamma-prime eutectic colonies were commonly associated with the fracture paths. An example of a typical creep rupture test specimen is shown in Figure 31a for the TRW-NASA VIA composition. A comparison with the microstructure shown in Figure 8 for the Series I powder metallurgy TRW-NASA VIA indicates the close similarity in structures, particularly the size and the distribution of the eutectic gamma-prime colonies. The major difference appears to be the fact that the metastable carbide alloy is somewhat smaller in overall grain size than the powder metallurgy alloy. In addition to the similarity in gamma-prime eutectic colonies, another feature common to the fracture surfaces of these three alloys was the general absence of porosity or voids.

The fracture paths and the general microstructure of the IN-100 test specimens was somewhat different than the other Series I metastable carbide alloys. An example of a typical structure is shown in Figure 31b. While it is apparent that the overall grain size for the IN-100 alloys was slightly smaller than the other alloys, a more important feature is the presence of isolated groupings or clusters of smaller grains (ASTM 8 or finer). These clusters of fine grains were sites for crack initiation. Their presence in the IN-100 alloy was attributed to the fact that localized inhomogeneities in carbide distribution remained after the attrition operation. This poor distribution was carried over into the HIP'ed structure. That these regions were not observed in the as-HIP'ed, or the heat treated material demonstrated their limited distribution in the alloy. There was little doubt, however, as to their ability to initiate cracks and degrade the mechanical properties.

The other important aspects of the microstructures of the failed test bar specimens were (1) the lack of observed concentrations of oxides along fracture surfaces or grain boundaries and (2) the presence of uncombined silicon in the grain boundaries of all four alloys. The absence of oxide particles along grain boundaries indicated that the high oxygen levels in the metastable carbide alloys did not result in embrittling oxide networks. That the higher oxygen levels could have contributed to the poor mechanical properties by more subtle mechanisms, however, could not be completely eliminated from consideration. The presence of silicon in the grain boundaries resulted from the decomposition of the metastable SiC particles during the HIP consolidation operation. The degree to which the uncombined grain boundary silicon contributed to the poor property levels was not established.



(a) TRW-NASA VIA



(b) IN-100

Figure 31. Light photomicrographs of Task III (metastable carbide) Series I TRW-NASA VIA and IN-100 creep-rupture test specimens. Magnification, 100X.

b. Series II Alloy Studies

(1) Alloy Formulation

For the Task III metastable carbide superalloys, the three aim compositions presented in Table XXVI were selected for evaluation in Series II. The desired chemistries are presented in terms of weight percent. These alloys were all modifications of TRW-NASA VIA, chosen as the base alloy for Series II because it exhibited the best overall combination of tensile and creep-rupture properties in the Series I mechanical property screening studies. Similar to the results for the powder metallurgy compositions, the predominantly intergranular failure mode of the metastable carbide TRW-NASA VIA alloy, Figure 31a, suggested that alloy modifications be directed towards improving creep-rupture capabilities primarily through grain boundary strengthening. In addition to this, however, there was another problem with the Series I metastable carbides involving the choice of SiC as the metastable carbide additive. During HIP consolidation, decomposition of the metastable SiC occurred and the carbon immediately recombined with titanium and tantalum to form stable carbides. These particular carbides inhibited grain growth during the subsequent heat treatment processing of these alloys. Therefore, in order to produce increased grain size and the desired discrete particle grain boundary carbide formation, VC, a more stable carbide than SiC, was used for the Series II alloys. VC was expected to improve mechanical properties in another regard. After SiC decomposition, it was observed that SiC had diffused to the grain boundaries where its presence there as a possible embrittling agent was suspect. Since vanadium is a gamma-prime forming element, the decomposition of VC was thus not expected to result in residual amounts of vanadium in the grain boundaries.

Compared to the base alloy composition shown in Table XXIII Modification I was aimed at the identical chemistry with the exception that VC replaced SiC as the metastable carbide. Note also that the high carbon aim was also retained in this alloy. Modification II featured increased levels of grain boundary strengthening elements compared to the Series I baseline alloy. The hafnium aim was increased from 0.43 to 2.5% and a rhenium addition was aimed at the 0.2% level. The carbon aim was also increased from 0.13 to 0.25% to explore the possible benefits of increased carbon content. As shown in the Series I alloys TRW-NASA VIA and TAZ-8A, high carbon in the metastable carbide alloys did not inhibit attaining a large grain size in the HIP'ed material. Modification III was identical to the Task I Modification III powder metallurgy alloy, reported to have a substantial creep life advantage over the conventional TRW-NASA VIA (48). It was included in Series II to compare the effects of metastable carbide processing with the powder metallurgy mechanical properties.

The actual compositions in weight percent of the material processed during the HIP parametric study are also listed in Table XXVI and represent material used for the mechanical property screening evaluations. Comparison of the aim compositions with the actual chemistries indicated that, with the exception of the carbon content for the Modification II alloy, the desired compositions were achieved. The carbon content for this alloy was lower than the aim level (0.18% versus 0.25%) but the composition was utilized for the Series II efforts because at this level it still represented the highest carbon content of the metastable carbide alloys. The higher vanadium contents of the Series II alloys compared to the silicon contents of the Series I alloys, Table XXIII, were a function of the increased carbon levels of the Series II alloys. The oxygen levels of both series of metastable carbide alloys remained high, reflecting the increased processing and handling required for these alloys.

Table XXVI

Task III (Metastable Carbide Alloys) Series II Alloy Compositions(1)

Alloy		C	Cr	Mo	Ti	Al	Co	W	Zr	B	Ta	Cb	Hf	Re	V	O_2	N_2
TRW-NASA VIA	Aim	0.13	6.1	2.0	1.0	5.4	7.5	5.8	0.05	0.01	9.0	0.5	0.43	0.20			
Modification I	Actual	0.12	5.8	2.02	1.1	5.4	7.6	5.9	0.07	0.011	8.8	0.65	0.66	0.21	0.37	268	37
TRW-NASA VIA	Aim	0.25	6.1	2.0	1.0	5.4	7.5	5.8	0.05	0.01	9.0	0.5	2.50	0.20			
Modification II	Actual	0.18	5.4	1.95	1.06	5.4	7.4	5.9	0.10	0.012	8.7	0.63	2.41	0.20	0.55	340	58
TRW-NASA VIA	Aim	0.10	6.1	1.0	2.0	4.5	10.0		0.05	0.01	6.0	2.0					
Modification III	Actual	0.08	5.9	0.95	2.1	4.4	10.3		0.07	0.012	5.95	1.95		0.28	360	58	

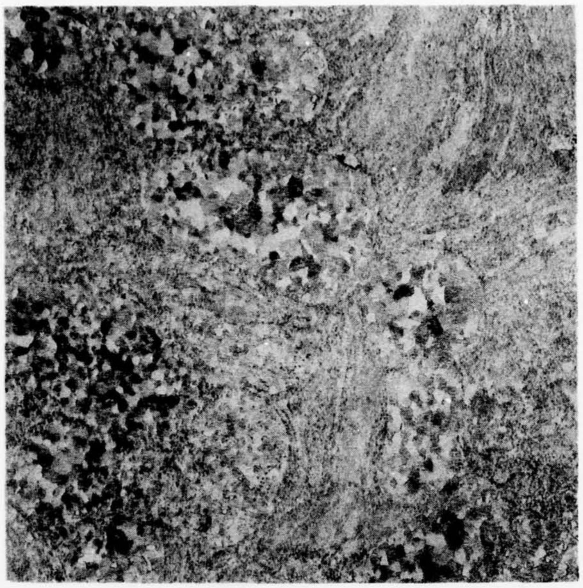
(1) In weight percent, except for oxygen and nitrogen, which are reported in parts per million.

(2) Attrition/HIP Parametric Study

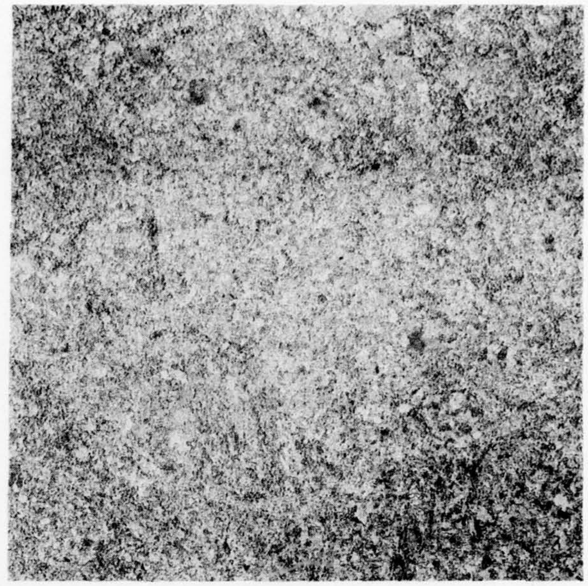
An attrition study was also conducted on the Series II alloys to establish the processing parameters required to achieve proper mixing of the VC particles with the carbon free base powders. Trial attrition processing was conducted on carbon free TRW-NASA VIA Modification I base powders to which were added the proper amounts of 3-4 micron or less VC particles to achieve a carbon content of 0.10 weight percent. Attrition was conducted on the powders using a ball/powder charge ratio of 16/1 under an argon atmosphere to minimize contamination of the powders. Attrition was conducted at 4 hours as a direct comparison to the Series I composition. The results of the trial indicated that iron, nitrogen and oxygen contents were all comparable to the Series I TRW-NASA VIA alloy also attrited for 4 hours. The carbon content was higher for the Series II Modification II trial alloy, while the vanadium content was higher than that of silicon in the Series I alloy, reflecting the aim for higher carbon content through VC additions for the Series II alloy. The overall appearance of the attrited particles was similar to that shown in Figure 29b for the Series I alloy. As a result of the attrition study, a time of 4 hours was selected for the Series II metastable carbide alloys.

A HIP parametric study was conducted on the Series II alloys in order to determine the processing conditions required to achieve complete densification in the consolidated metastable carbide powders. The initial HIP condition investigated for each alloy included 1304°C(2380°F)/103.4 MPa (15 ksi) for four hours and was chosen in an attempt to densify the powders without decomposing the metastable VC particles. Since the Series I work indicated that HIP at 1316°C (2400°F) resulted in decomposition of SiC, it was reasoned that HIP consolidation of a more stable carbide at a lower temperature would not cause decomposition of the metastable carbide. Analysis of the as-HIP'ed microstructures indicated that complete densification was achieved at the 1304°C (2380°F) temperature without decomposition of the metastable VC carbides. As shown in the photomicrographs of Figure 32, a fine carbide dispersion was obtained throughout the as-HIP'ed microstructures. Microprobe analysis identified these particles as vanadium rich carbides. Little evidence of grain growth was observed as a result of this HIP operation, however, with grain sizes ranging from ASTM 8-9 or lower.

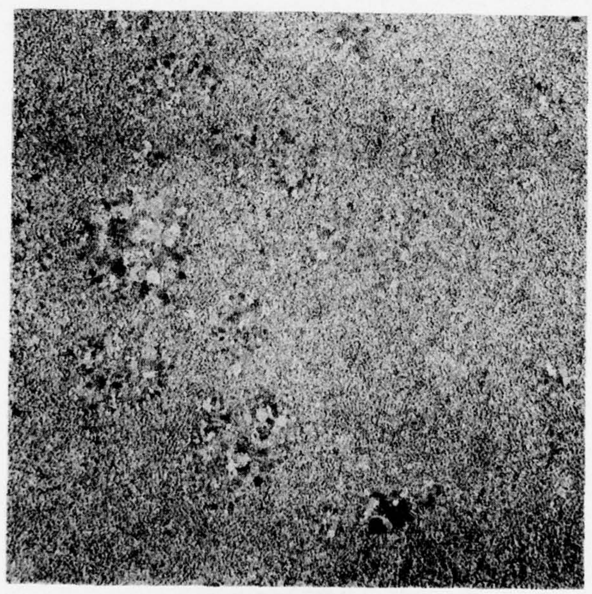
Heat treatment of these alloys at 1316°C (2400°F)in order to solution the VC particles and produce grain growth was largely unsatisfactory because of the degree of incipient melting observed throughout the microstructures. While partial carbide solutioning was achieved, and grain growth to approximately ASTM 3-5 was obtained, the microporosity associated with the incipient melting throughout the structure indicated that heat treatment to still higher temperatures would result in unacceptable porosity levels. To explore the possibility of achieving these grain sizes without the accompanying microporosity, material HIP'ed at 1304°C (2380°F) was re-HIP'ed at 1316°C (2400°F). The microstructures of the Series II metastable carbide alloys after the second HIP operation are shown in Figure 33. Comparison with the photomicrographs shown in Figure 32 for material HIP'ed at 1304°C (2380°F) indicate that appreciable vanadium carbide solutioning was achieved with an accompanying ASTM 3-5 grain size. While some evidence of incipient melting was observed, there was no porosity associated with these particular areas. These grain sizes were, in general, comparable to that for the Series I metastable carbide TRW-NASA VIA composition, Figure 31a.



(a) TRW-NASA VIA
Modification I

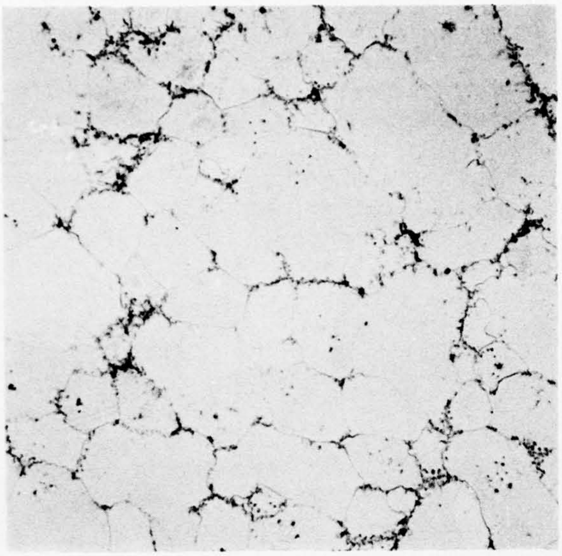


(b) TRW-NASA VIA
Modification II

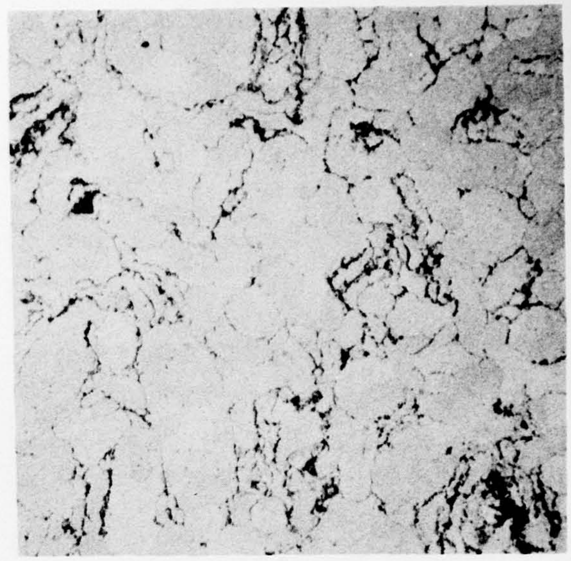


(c) TRW-NASA VIA
Modification III

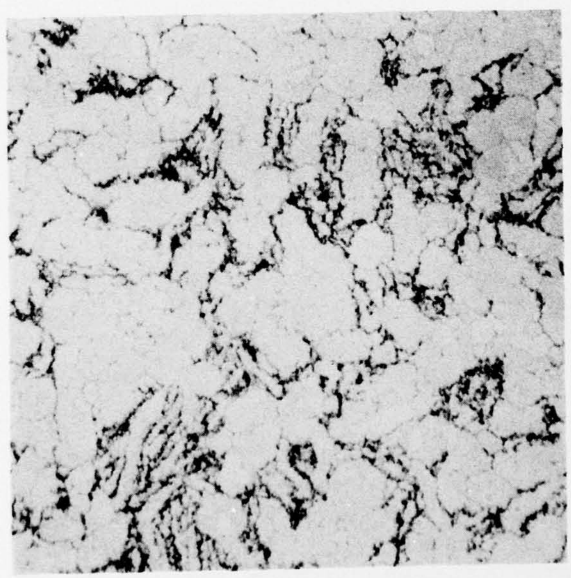
Figure 32. Light photomicrographs of Task III (metastable carbide) Series II alloys after HIP at 1304°C (2380°F)/103.4 MPa (15 ksi)/4 hours. Magnification, 100X



(a) TRW-NASA VIA
Modification I



(b) TRW-NASA VIA
Modification II



(c) TRW-NASA VIA
Modification III

Figure 33. Light photomicrographs of Task III (metastable carbide) Series II alloys after HIP at 1304°C (2380°F)/103.4 MPa (15 ksi)/4 hours and 1316°C (2400°F)/103.4 MPa (15 ksi)/4 hours. Magnification, 100X.

As a result of this HIP consolidation study, two different compaction operations were selected for the Series II mechanical property studies. The single step consolidation at 1304°C (2380°F) was selected to evaluate the mechanical properties of materials exhibiting a fine carbide distribution throughout the microstructure. The two step consolidation operation was selected because the resultant grain sizes of the various alloys were comparable to the Series I base alloy. For both cases it was decided to apply the same 24 hour 899°C (1650°F) carbide stabilization aging treatment utilized for the Task I powder metallurgy alloys and the Series I metastable carbide alloys. This allowed direct comparisons to be made between the various compositions.

(3) Mechanical Property Screening Evaluation

(a) Tensile Test Results

Tensile tests were conducted at 927°C (1700°F) on three specimens from each of the Series II metastable carbide alloys. A single specimen was HIP consolidated at 1304°C (2380°F) and given the 24 hour age, while duplicate specimens were double consolidated and given the 24 hour age. The results of these tests are listed in Table XXVII. These tensile data were significant not in terms of comparisons between the three Series II alloys themselves, but rather, in terms of the comparison between the Series I TRW-NASA VIA tensile data, Table XXIV, and the Series II data. This comparison indicated that as a group, the Series II alloys were inferior to the base TRW-NASA VIA composition evaluated in Series I. The Series II alloys HIP consolidated at 1304°C (2380°F), in particular, exhibited poor strength and ductility properties in comparison to the Series I alloy as well as compared to double consolidated material. In all instances, for example, the test specimens failed before 0.2% offset yield was reached. The double consolidated material was somewhat better, especially the Modification II and III alloys, which exhibited an average yield strength of approximately 434.4 MPa (63 ksi). This value, however, was appreciably below the 517 MPa (75 ksi) average exhibited by the Series I alloy. A plot of the yield strength for the Series II metastable carbide alloys compared to the Series I TRW-NASA VIA alloy is shown in Figure 34, and all of the yield strength data values were included in this plot for each of the alloys. While Modifications II and III exhibited the best yield strengths of the Series II alloys, it was apparent that the chemistry and processing changes made in these alloys did not result in improvements compared to the Series I alloy. This indicated that none of the Series II alloys offered potential for further alloy developments for the Series III effort.

(b) Creep-Rupture Test Results

Creep-rupture tests were conducted at 954°C (1750°F)/206.8 MPa (30 ksi) on five specimens from each of the Series II alloys and the results of these tests are listed in Table XXVIII. Specimens prepared by single step and double step consolidation operations were both included in the evaluation. The results were similar to the tensile tests in that as a group, the Series II metastable carbide alloys were significantly inferior to the Series I TRW-NASA VIA composition. The Series I specimens exhibited an average rupture life of 25.6 hours, while Modification III, the best of the metastable carbide Series II alloys, exhibited an average rupture life of approximately 5 hours. The Modification I and II alloys all exhibited failure times of one hour or less. The times to reach 0.1% creep were also poor for these alloys, with none of the specimens

Table XXVII

Task III (Metastable Carbide Superalloys) Series II 927°F (1700°F) Tensile Test Results

Alloy	Processing Condition	Ultimate Tensile Strength MPa	Ultimate Tensile Strength Ksi	Yield Strength MPa	Yield Strength Ksi	% Elongation	% Reduction Area
TRW-NASA VIA Modification I	HIP Condition 1 Plus Age (1)	311.7	45.2 (2)		(3)	0.3	0.0
	HIP Condition 2 Plus Age	343.4	49.8 (2)	310.3	45.0	0.5	0.3
		356.5	51.7 (2)	291.0	45.2	0.5	0.3
TRW-NASA VIA Modification II	HIP Condition 1 Plus Age	231.7	33.6		(3)	0.2	0.2
	HIP Condition 2 Plus Age	560.6	81.3	427.5	62.0	0.2	0.2
		458.9	79.6 (2)	437.9	63.5	0.5	0.1
TRW-NASA VIA Modification III	HIP Condition 1 Plus Age	522.7	75.8		(3)	0.3	0.8
	HIP Condition 2 Plus Age	607.4	88.1 (2)	427.5	62.0	3.4	1.9
		589.6	85.5	450.3	65.3	3.1	3.1

Air Force Program Goal: 517 MPa (75 Ksi) offset yield strength at 927°C (1700°F) in a section size of 2.8 cm (7 inches).

HIP Conditions

1. 1304°C (2380°F)/103 MPa (15 Ksi)/4 Hrs.
2. 1304°C (2380°F)/103 MPa (15 Ksi)/4 Hrs. Plus 1316°C (2400°F)/103 MPa (15 Ksi)/4 Hrs.

(1) Age 899°C (1650°F)/24 Hrs/Air Cool.
(2) Thread Failure - Ductility Measured in Gage Section.
(3) Specimen Failed Before 0.2% Offset Yield was Reached.

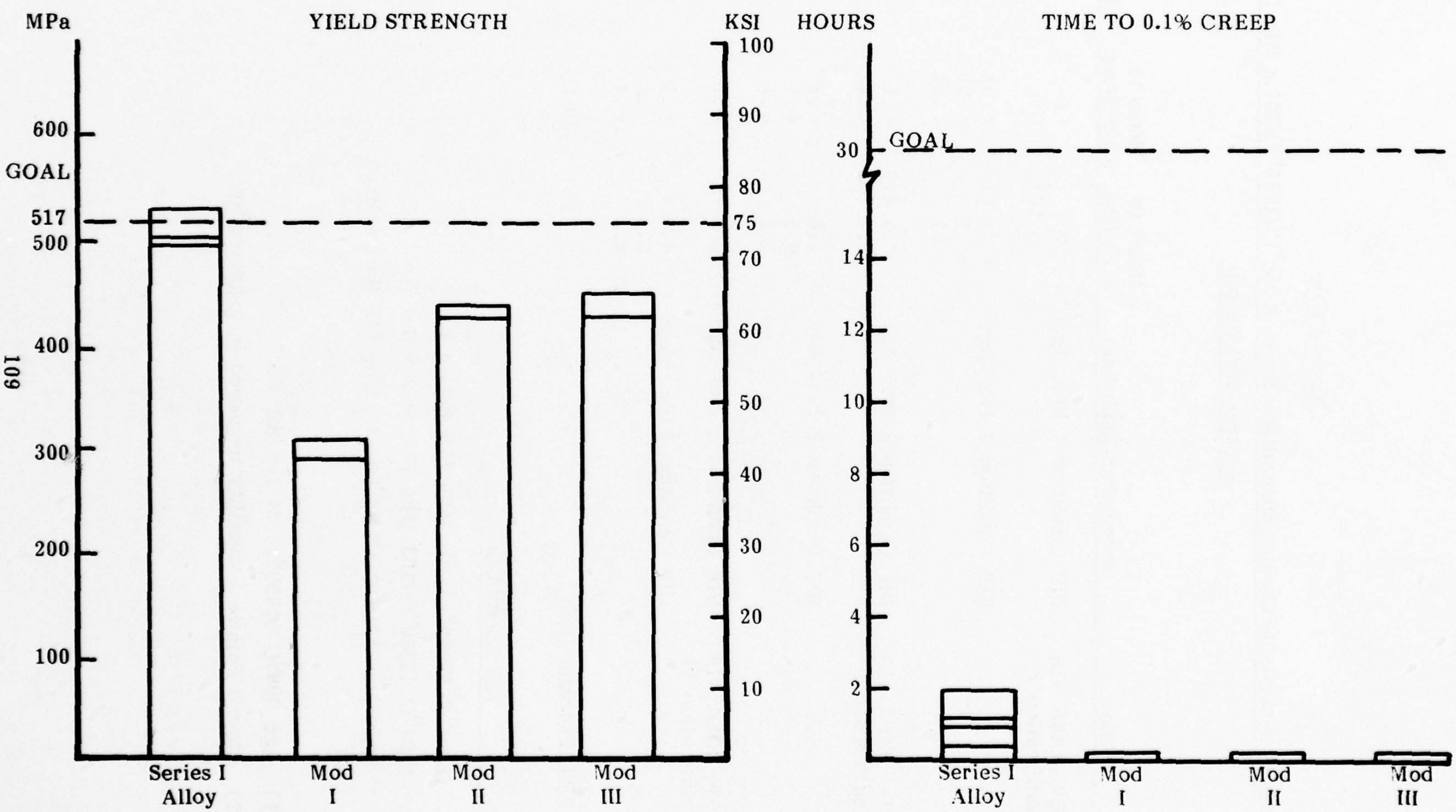


Figure 34. Yield strength and time to 0.1% creep for Task III (metastable carbide) Series II alloys compared to Series I TRW-NASA VIA alloy.

Table XXVIII

Task III (Metastable Carbide Superalloys) Series II 954°C (1750°F)/206.8 MPa (30 Ksi) Creep Rupture Test Results

Alloy	Processing Condition	Hours to Failure	Hours to 0.1% Creep	% Elongation	% Reduction Area
TRW-NASA VIA Modification I	HIP Condition 1 Plus Age(1)	0	0	0.7	0
		0 (2)	0	0	0
	HIP Condition 2 Plus Age	< .1 (2)	< .01	1.7	0.6
		< .1	< .01	1.3	0
		<.1	< .01	0.5	0
TRW-NASA VIA Modification II	HIP Condition 1 Plus Age	0.3 (2)	< .1	0.3	0
		0.1 (2)	< .1	0.0	0
	HIP Condition 2 Plus Age	1.0	< .1	1.0	0
		0.5	< .1	1.2	0.6
		1.1	< .1	1.5	0.6
TRW-NASA VIA Modification III	HIP Condition 1 Plus Age	2.6	< .1	0.7	0
	HIP Condition 2 Plus Age	4.6	< .1	3.5	2.5
		5.8	< .1	1.7	3.1
		4.8	< .1	3.0	2.4
AFML Program Goal			30.0		

HIP Conditions

1. 1304°C (2380°F)/103 MPa (15 Ksi)/4 Hrs
2. 1304°C (2380°f)/103 MPa (15 Ksi)/4 Hrs
   plus 1316°C (2400°F)/103 MPa (15 Ksi)/4 Hrs

(1) Age 899°C (1650°F)/24 Hrs/Air Cool

(2) Thread Failure - ductility measured in gage section

exceeding 0.1 hours. A plot of all of the test values of time to reach 0.1% creep for the Series II alloys compared to the Series I base composition is shown in Figure 34. It was evident that the alloy modifications employed for the Series II study did not exhibit potential for further development in the Series III effort.

(c) Metallographic Analysis

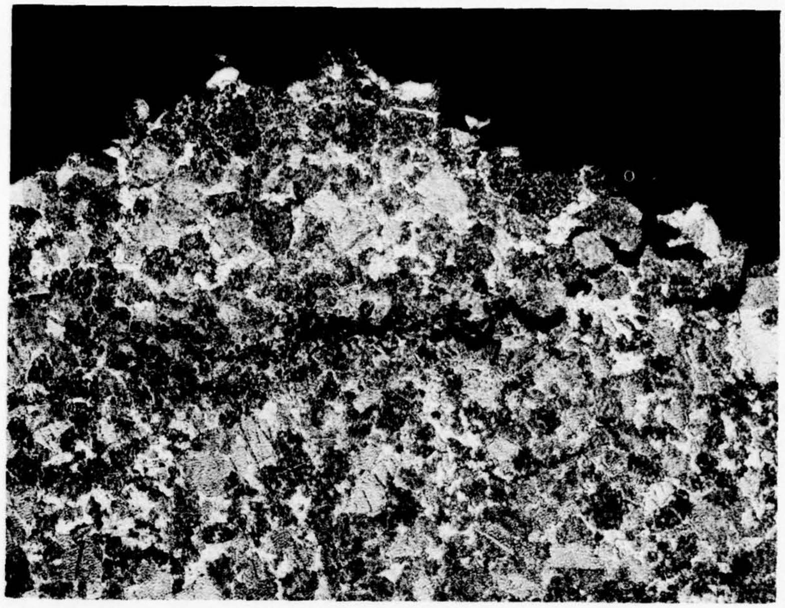
Metallographic analysis was conducted upon failed test specimens and indicated that a predominantly intergranular failure mode was common for the three Series II alloys for both the tensile and the creep-rupture tests. For the various alloys the fracture paths were dependent upon the consolidation operations used to prepare test specimens. In those test specimens consolidated in the single step operation at 1304°C (2380°F), the fracture paths were usually associated with the isolated clusters of fine recrystallized grains (ASTM 8-9 or finer) within the generally unrecrystallized matrix. Examples of these clusters of recrystallized grains are shown in Figure 32 for material in the as-HIP'ed condition. Cracks were observed to nucleate within these clusters and propagate through the unrecrystallized matrix. It was apparent that the fine distribution of carbides resulting from the low temperature HIP operation contributed little to the elevated temperature tensile and creep rupture properties of these compositions.

In those specimens consolidated with the double HIP operation at 1304°C (2380°F) and 1316°C (2400°F), the fracture paths were considerably different. In these instances fractures were usually associated with the remnant carbide networks resulting from the HIP operation. These networks can clearly be seen in the photomicrographs of as-HIP'ed material shown in Figure 33, and were generally found to be rich in vanadium. Typical examples of the fracture surfaces of creep rupture test specimens are shown in Figure 35 for the Modification II and III alloys. These photomicrographs indicate that in addition to the carbide networks the Series II metastable carbide alloys also exhibited an inhomogeneous microstructure in terms of grain size. Comparison of the grain sizes in the as-HIP'ed material, Figure 33, with those of the actual test bar specimens indicated a slightly finer grain size in certain regions of the failed bars. It was apparent that the distribution of the carbide networks was largely responsible for the variations in grain size throughout the specimens. The grain sizes tended to be finer in those areas relatively heavy and continuous with carbides.

c. Series III Alloy Studies

(1) Alloy Formulation

For the Task III metastable carbide superalloys, the two aim compositions presented in Table XXIX were selected for evaluation in Series III. The desired chemistries are presented in terms of weight percent. Analysis of the mechanical property results of the Series II metastable carbide alloys indicated that none of the three compositions offered potential for further alloy development in Series III. Certain processing modifications, however, were suggested by the results, including changes to minimize the formation of vanadium carbide networks throughout the microstructure. Because of these considerations, the Series III compositions represent not so much variations based upon the chemistry of the Series II alloys, but rather a combination of



(a) TRW-NASA VIA
Modification II

(b) TRW-NASA VIA
Modification III

Figure 35. Light photomicrographs of Task III (metastable carbide) Series II alloys showing intergranular failure mode typical of creep-rupture test specimens. Magnification, 100X.

Table XXIX

Task III (Metastable Carbide Alloy) Series III Alloy Compositions[1]

Alloy		C	Cr	Mo	Ti	Al	Co	W	Zr	B	Ta	Cb	Hf	Re	V	O_2	N_2
TRW-NASA VIA	Aim	0.25	9.0	2.0	0.5	5.4	7.5	5.8	0.05	0.01	6.0		2.0	0.20			
Modification IV	Actual	0.22	9.31	1.97	0.38	5.52	7.36	5.84	0.05	0.009		1.88	0.19	0.56	470	49	
TRW-NASA VIA	Aim	0.04	6.1	2.0	1.0	5.4	7.5	5.8	0.05	0.01	9.0	0.5	1.5	0.02			
Modification V	Actual	0.03	5.77	1.89	1.01	5.41	7.41	5.74	0.09	0.015	8.9	.97	0.18	0.25	430	45	

(1) In weight percent, except for oxygen and nitrogen, which are reported in parts per million.

chemistry modifications suggested by the Series I results as well as those of the Task I powder metallurgy alloys. The primary contribution of the results of the Series II metastable carbide alloys was the processing modifications employed for the Series III alloys. Because of the relative stability of the VC metastable carbide additives in comparison to the SiC particles of Series I, VC was elected for the carbon additions for the Series III efforts.

The basic aim of the compositions selected for study in the Series III evaluation was directed towards minimizing the stable carbide networks which contributed to mechanical property degradation in Series II. In order to accomplish this, Modification IV was designed to include reduced tantalum (6 versus 9%) and titanium (0.5 versus 1.0%) compared to the Series I composition. Columbium was also dropped from this composition. It was anticipated that these changes would result in minimizing the stable carbide network formed in the Series II alloys upon the partial solutioning of the metastable VC particles. Modification IV also featured an increase in chromium from 6 to 9% compared to the Series I alloy to promote the formation of $M_{23}C_6$ type grain boundary carbides. The carbon level for this alloy was aimed at 0.25% and the hafnium was aimed at 2.0%. The Modification V alloy featured a carbon aim of 0.04% compared to the 0.10% or higher levels of the other metastable carbide alloys in an attempt to reduce the carbide networks through reduced carbon content. The hafnium level for this composition was aimed at 1.5%, representing a median level between the Series I alloy and the Modification IV composition.

The actual compositions in weight percent of the material processed during the HIP parametric study are also listed in Table XXIX and are representative of material used for the mechanical property screening evaluations. Comparison of the aim compositions with the actual chemistries indicated that, with the exception of the hafnium content for the Modification V alloy, the desired compositions were achieved. The hafnium content for this alloy was lower than the aim level (0.97 versus 1.5%) but the composition was utilized for the Series III efforts because at this level the hafnium content still represented an intermediate level between the Series I composition (0.51%) and the Modification IV alloy (1.88%). It was also observed that the overall oxygen contents of the Series III alloys were somewhat higher than the Series I and II compositions. The Series III oxygen levels were all above the 400 ppm level, while those for Series I and II were below 360 ppm. As will be discussed in more detail in the attrition study, this reflected the increased attrition time used to blend the powders with a minimum remnant carbide network throughout the microstructure.

(2) Attrition/HIP Parametric Study

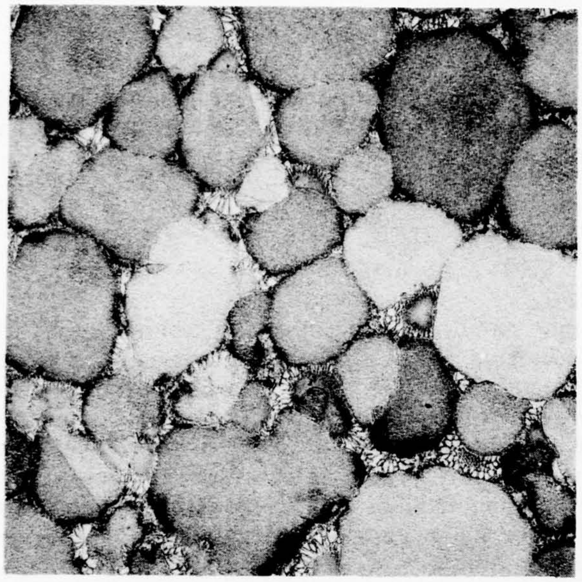
An attrition study was conducted on the Series III alloys to establish the processing parameters required to achieve proper mixing of the VC particles without the attendant stable carbide networks prevalent in the Series II alloys. Initial attrition processing was conducted on carbon-free TRW-NASA VIA Modification V base powders to which were added the proper amounts of 3-4 micron or less VC particles to achieve a carbon content of 0.15% by weight. Attrition was conducted on the powders using a ball/powder charge ratio of 16/1 under an argon atmosphere for 20 hours in an effort to disperse the carbide networks shown in Figure 33 for as-HIP'ed Series II alloys.

The results of this trial are indicated below in terms of iron, nitrogen, oxygen, carbon and vanadium contents. Iron, carbon and silicon are given in weight percent while nitrogen and oxygen are given in parts per million (ppm).

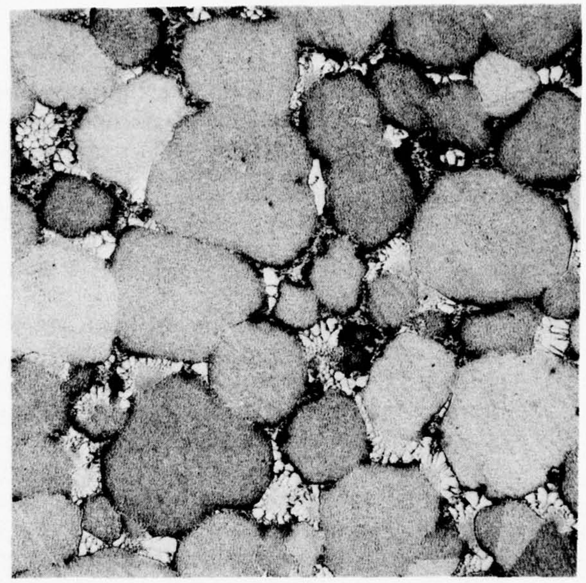
Iron	Nitrogen	Oxygen	Carbon	Vanadium
.089	56	435	0.16	0.48

It was observed that the 20 hour attrition time resulted in the desired carbon content in the powders. Oxygen and nitrogen contamination both increased compared to that obtained for material attrited for 4 hours in Series I and II, as was to be expected for the increased attrition time. Iron pick-up, however, was not significantly higher than the approximately 0.08% level obtained for the Series I and II alloys. The vanadium level was comparable to that observed for Modification II alloy, Table XXVI, which exhibited a somewhat similar carbon content. Metallographic analysis of the as-attrited particles indicated an appearance similar to the particles shown in Figure 29 for the attrited Series I alloy with the exception that significantly larger numbers of the particles (approximately 90%) were completely worked and retained little of their as-atomized dendritic structure. This suggested that sufficient blending of the metastable VC particles had been achieved with the carbon-free base powders to minimize the carbide networks characteristic of the Series II alloys. On the basis of this consideration as well as the fact that a five fold increase in attrition time resulted in an increase of only approximately 70-100 ppm oxygen pick-up in the attrited powders, the 20 hour attrition time was selected for the Series III metastable carbide alloys.

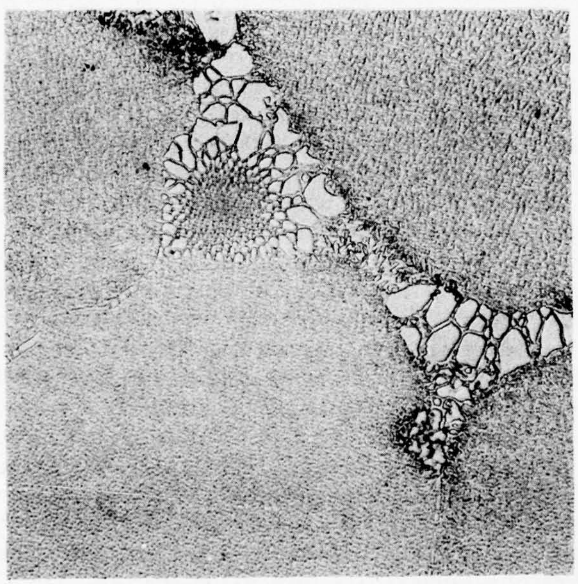
A HIP parametric study was conducted on the Series III alloys in order to determine the processing conditions required to achieve complete densification in the consolidated metastable carbide superalloys without the attendant networks of stable carbides. The initial HIP condition investigated for each alloy included 1316°C (2400°F) for four hours and was chosen on the basis of the Series II work which indicated partial VC solutioning at this temperature as well as an ASTM grain size ranging from 3-5. Metallographic analysis of the microstructures of the Series III metastable carbide alloys after the HIP operation indicated a greater degree of homogeneity in terms of grain size than was apparent in the Series II alloys. This increase in homogeneity was attributed to the 20 hour attrition time compared to the 4 hour attrition time of the first two series. Modification IV and V alloys both evidenced partial VC solutioning as well as an ASTM 2-4 grain size. While thermal exposures to temperatures higher than 1316°C (2400°F) resulted in more complete VC solutioning, appreciable amounts of incipient melting were observed throughout the microstructure. As a result, it was decided to use 1316°C (2400°F) as the consolidation temperature for the Series III alloys. Since little further VC modification could be achieved without the accompanying incipient melting, it was decided to employ the 24 hour carbide stabilization treatment at 899°C (1650°F) for the Modification IV and V alloys. Examples of the microstructures of the Series III alloys after HIP at 1316°C (2400°F) and the 24 hour age at 899°C (1650°F) are shown in Figure 36. The overall structures of these alloys are similar to that presented in Figure 31a for the Series I TRW-NASA VIA composition. The grain sizes of the alloys are comparable as are the distributions of the gamma-prime eutectic colonies. The individual platelets within the colonies of the Series III alloys are somewhat better defined than those appearing in the Series I alloy, and could reflect the higher hafnium content of the Modification IV and V alloys. This effect was observed with the Task II casting superalloys.



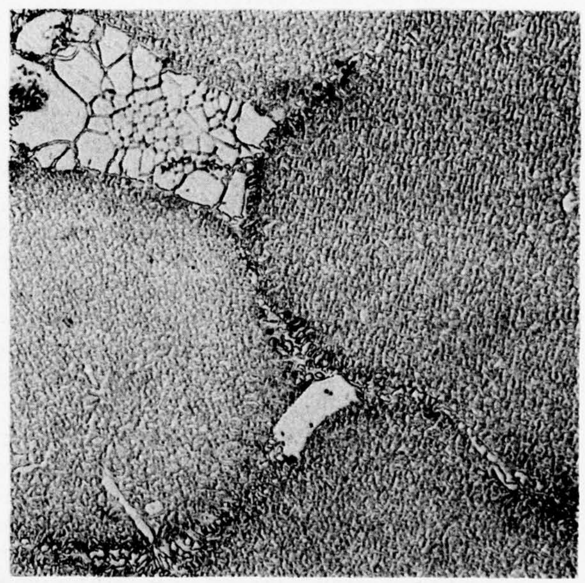
(a) Modification IV, 100X



(b) Modification V, 100X



(c) Modification IV, 500X



(d) Modification V, 500X

Figure 36. Light photomicrographs of Task III (metastable carbide alloys) Series III TRW-NASA VIA Modifications IV and V after HIP at 1316^{o}C (2400^{o}F)/103.4 MPa (15 ksi)/4 hours plus 24 hours age at 899^{o}C (1650^{o}F).

(3) Mechanical Property Characterization

(a) Tensile Test Results

Tensile tests were conducted on five specimens from each of the Series III metastable carbide alloys. Initially, duplicate specimens were tested at 927°C (1700°F). When comparison of the results with those for the series I TRW-NASA VIA alloy indicated little improvement in ultimate tensile strength or 0.2% yield strength, the remaining three specimens were tested at other temperatures within the range of isothermal forging conditions to provide more information as to the potential capability of the metastable carbide alloys to perform as isothermal forging die materials (5). For these purposes, duplicate tests were conducted at 830°C (1525°F) and a single test was conducted at 760°C (1400°F). The results of these tensile tests are listed in Table XXX.

The tensile test results at 927°C (1700°F) indicated that while both Modification IV and V alloys exhibited comparable 0.2% yield strengths, their average yield strength values were both inferior to that for the Series I TRW-NASA VIA composition and were also below the program goal of 517 MPa (75 ksi). Both alloys were characterized by yield strengths of approximately 434.4 MPa (63 ksi), compared to the average yield strength of 517 MPa (75 ksi) for the Series I alloy. In addition to the poor yield strength values, both of the Series III alloys were characterized by extreme notch sensitivity in that thread failures occurred in all four test specimens at 927°C (1700°F). Because of this, neither percent elongation nor reduction of area were reported for these alloys. A plot of the yield strength for the Series III metastable carbide superalloys compared to the Series I TRW-NASA VIA alloy and the Modification II composition (the best of the Series II alloys) is shown in Figure 37. All of the yield strength data values were included in this plot for each of the alloys. It is evident from this plot that the Series III alloys were comparable in yield strength to the Modification II alloy, and that the Series I TRW-NASA VIA alloy exhibited the highest yield strength values of the metastable carbide alloys.

Analysis of the Series III results for the 830°C (1525°F) and 760°C (1400°F) tensile tests indicated some inconsistency in terms of which alloy exhibited superior tensile properties at a particular test temperature. The Modification IV alloy offered slightly superior yield strength at 830°C (1525°F), while Modification V offered significantly improved yield strength at 760°C (1400°F). It must be pointed out, however, that only a single specimen was tested at 760°C (1400°F), while duplicate tests were conducted at 830°C (1525°F). In spite of these considerations the implications of these yield strength differences between the alloys were relatively insignificant in view of the fact that, as a group, the Series III metastable carbide superalloys exhibited inferior tensile properties compared to published values for the base TRW-NASA VIA composition in the cast form. As a comparison, for example, Modification IV exhibited an average yield strength of 639.9 MPa (92.8 ksi) at 830°C (1525°F), the highest of the Series III metastable carbide alloys. Published values for cast TRW-NASA VIA indicate an approximate yield strength of 827.4 MPa (120 ksi) at this temperature (59). At 769°C (1400°F), Modification V exhibited a yield strength of 844 MPa (122.4 ksi), the best of the Series III alloys, compared to the published value of 951.5 MPa (138 ksi) for cast TRW-NASA VIA. While it is recognized that the published values for cast TRW-NASA VIA were obtained from cast-to-size test specimens, and as such a strong section size effect may be expected, the fact remains that the metastable carbide properties were still appreciably

Table XXX

Task III (Metastable Carbide Superalloys) Series III Tensile Test Results[1]

Alloy	Test Temperature °C	Test Temperature °F	Ultimate Tensile Strength MPa	Ultimate Tensile Strength Ksi	0.2% Offset Yield Strength MPa	0.2% Offset Yield Strength Ksi	% Elongation	% Reduction Area
TRW-NASA VIA	927	1700	630.4	90.7	453.1	65.7	(2)	(2)
Modification IV	927	1700	627.4	91.0	432.4	62.7	(2)	(2)
	830	1525	886.7	128.6	655.7	95.1	6.5	12.7
	830	1525	852.3	123.6	623.3	90.4	3.9	6.8
	760	1400	1040.4	152.2	728.8	105.7	14.6	17.9
TRW-NASA VIA	927	1700	453.1	65.7	445.5	64.6	(2)	(2)
Modification IV	927	1700	421.3	66.1	417.9	60.6	(2)	(2)
	830	1525	793.6	115.1	621.2	90.1	2.5	3.1
	830	1525	760.5	110.3	608.1	88.2	4.0	6.3
	760	1400	1007.0	146.0	844.0	122.4	2.8	5.0

Air Force Program Goal: 517 MPa (75 Ksi) 0.2% offset yield strength at 927°C (1700°F) in a section size of 2.8 cm (7 inches).

(1) Processing conditions for all specimens included HIP at 1316°C (2400°F)/103 MPa (15 Ksi)/4 hours plus 899°C (1650°F)/24 hours/air cool heat treatment.

(2) Thermal Failure

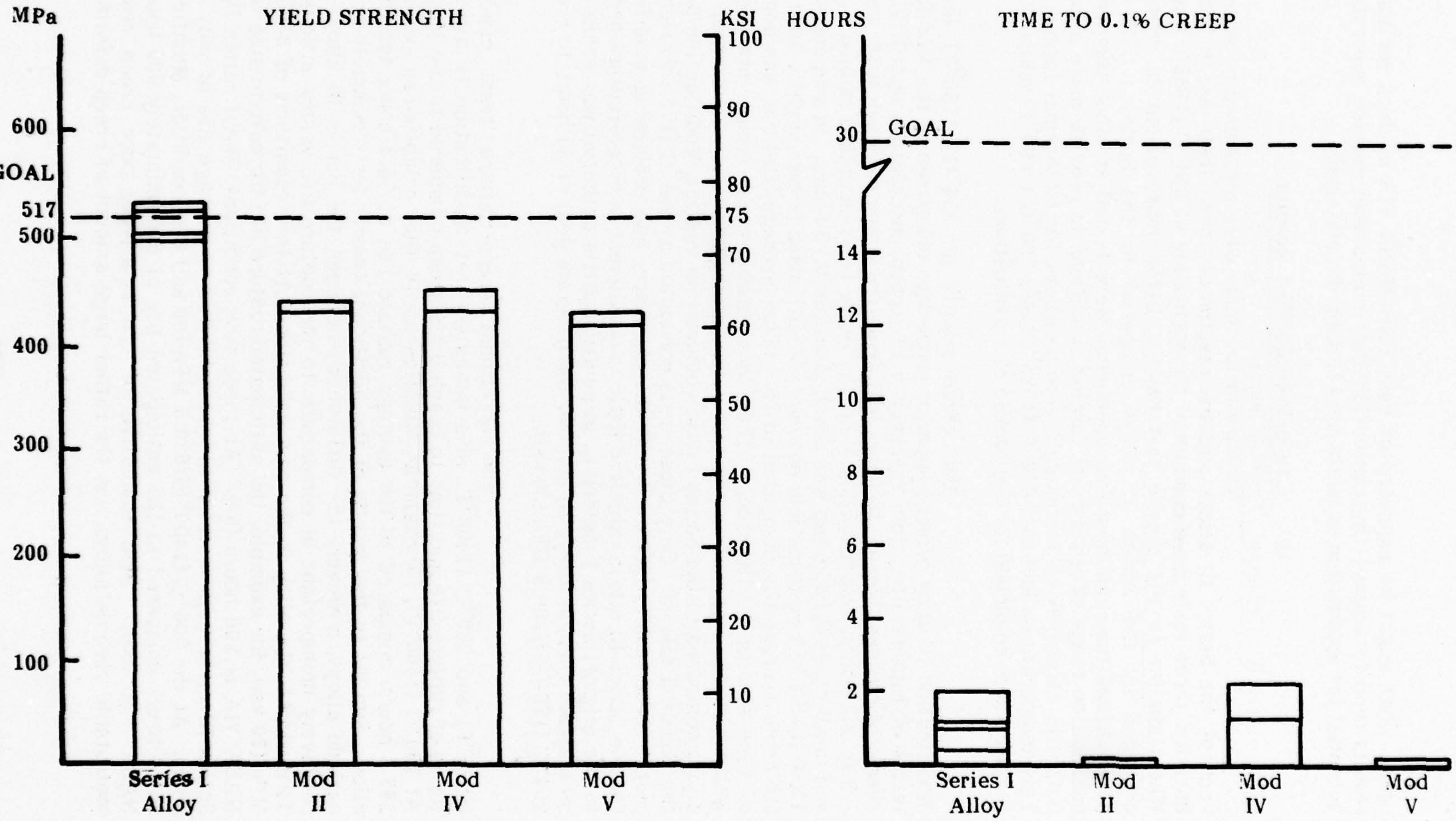


Figure 37. Yield strength and time to 0.1% creep for the Task III (metastable carbide) Series III alloys compared to Series I TRW-NASA VIA and Series II Modification II alloys.

below what might be expected of cast TRW-NASA VIA in thick sections. These tensile results tend to support the contention that metastable carbide superalloys exhibit little potential for application as isothermal forging die materials.

(b) Creep-Rupture Test Results

Creep-rupture tests were conducted on seven specimens from each of the Series III alloys and the results of these tests are listed in Table XXXI. Initially, three tests were conducted for each alloy at 954°C (1750°F)/206.8 MPa (30 ksi). When, similar to the tensile test results, little improvement in creep resistance was exhibited by the Series III alloys compared to the Series I TRW-NASA VIA base composition, the remaining four specimens were tested at other temperatures within the anticipated range of isothermal forging conditions to provide more information as to the potential capability of metastable carbide alloys for isothermal forging die applications. Test temperatures included 830°C (1525°F) and 760°C (1400°F) and were identical to the temperatures employed for the tensile test evaluations.

The creep results at 954°C (1750°F) indicated that the Modification IV alloy offered superior properties compared to the Modification V alloy in terms of rupture life, time to reach 0.1% creep and rupture ductility. Modification IV averaged rupture lives of 16 hours and exhibited times to reach 0.1% creep ranging from 1.9 to 2.3 hours. Modification V on the other hand exhibited failure lives below 4 hours, and in all cases 0.1% creep was reached within 0.20 hours. In comparison to the Series I TRW-NASA VIA composition, however, Modification IV was slightly inferior in rupture life (16 hours average life compared to 25.6 hours average life) but was comparable in terms of time to reach 0.1% creep. These comparable creep results are shown in Figure 37, which includes a plot of all the test values of time to reach 0.1% creep for the Series III alloys compared to the Series I base composition and the Modification II alloy (the best of the Series II alloys). Only those tests conducted at 954°C (1750°F) were included in this plot. These results indicated that the chemistry and processing modifications employed for the Series III alloys exhibited little improvement in creep-rupture properties compared to the original Series I material. In addition, little potential was exhibited by any of the metastable carbide alloys to achieve the program goal of 30 hours to reach 0.1% creep at 954°C (1750°F)/206.8 MPa (30 ksi).

The additional creep-rupture tests conducted at 830°C (1525°F) and 760°C (1400°F) also indicated that Modification IV alloy was superior in terms of rupture life and time to reach 0.1% creep compared to the Modification V alloy. At 760°C (1400°F), for example, Modification IV alloy exhibited an average rupture life of 265.8 hours, compared to the average rupture life of 74.3 hours for the Modification V alloy. Similar to the results of the additional tensile tests conducted on the metastable carbide alloys, however, the differences between the Series III alloys themselves were relatively unimportant in comparison to the rupture life values anticipated for the base TRW-NASA VIA alloy in the cast condition. At test conditions of 830°C (1525°F)/482.6 MPa (70 ksi), for example, the anticipated rupture life for cast-to-size test bars of TRW-NASA VIA is 200 hours (59). At these test conditions, Modification IV, the best of the Series III metastable carbide alloys, exhibited an average life of only approximately 65 hours. At the 760°C (1400°F)/620.5 MPa (90 ksi) test condition, Modification IV averaged 265.8 hours compared to the anticipated life of approximately 600 hours for cast TRW-NASA VIA (59). Also indicative of the relatively poor creep resistance of these metastable carbide alloys was the rather large amount of creep deformation observed at

Table XXXI

Task III (Metastable Carbide Alloys) Series III

Creep Rupture Test Results [(1)]

Alloy	Test Temperature ^{o}C	^{o}F	Stress Level MPa	Ksi	Hours to Failure	Hours to 0.1% Creep	% Elongation	% Reduction Area
TRW-NASA VIA	954	1750	206.8	30	21.4	2.3	5.2	2.0
Modification IV	954	1750	206.8	30	10.8	1.3	6.5	2.4
	954	1750	106.8	30	15.8	1.9	4.9	1.9
	830	1525	482.6	70	58.2	4.8	4.2	5.0
	830	1525	482.6	70	72.1	7.6	3.9	4.8
	760	1400	620.5	90	313.5	11.7	5.5	6.8
	760	1400	620.5	90	218.0	8.6	3.0	3.2
TRW-NASA VIA	954	1750	206.8	30	3.5	0.20	2.5	5.7
	954	1750	206.8	30	3.2	0.17		
	954	1750	206.8	30	(2)			
	830	1525	482.6	70	52.0	1.13	2.9	2.7
	830	1525	482.6	70	36.0	2.7	1.6	0.5
	760	1400	620.5	90	55.6	3.5	1.8	0.6
	760	1400	620.5	90	93.0	4.2	1.0	0.9
AFML Program Goal						30		

(1) Processing included HIP consolidation at 1316°C (2400°F)/103.4 MPa (15 Ksi)/4 hours plus heat treatment of 899°C (1650°F)/24 hours/air cool.

(2) Specimen failed on loading.

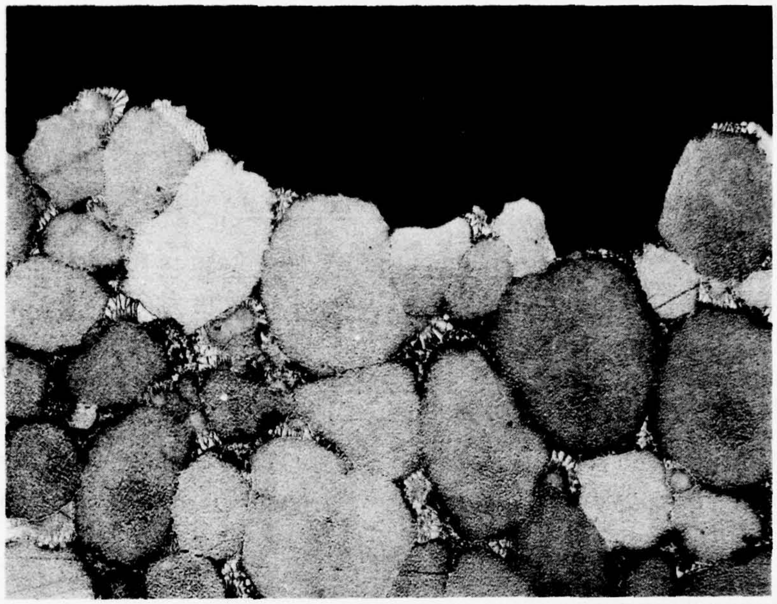
760°C (1400°F). Modification IV exhibited times to reach 0.1% creep ranging from 8.6-11.7 hours. This amount of creep deformation at 760°C (1400°F) was considered excessive compared to other types of nickel-base superalloys tested at this temperature (60).

(c) Metallographic Analysis

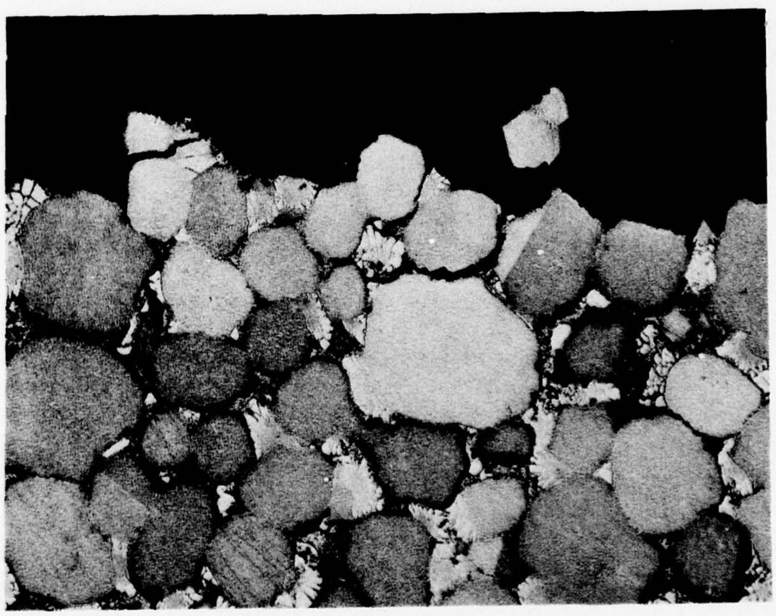
Metallographic analysis was conducted upon failed test bar specimens of the two Series III alloys evaluated in both the tensile and the creep-rupture tests. Only those tensile specimens tested at 927°C (1700°F) and those creep-rupture specimens tested at 954°C (1750°F) were examined in this effort to provide a direction comparison with the Series I and Series II metastable carbide results. The analyses indicated that an intergranular failure mode was typical for both the tensile and the creep-rupture tests. Typical examples of intergranular fracture paths are shown in Figure 38 for 954°C (1750°F) creep-rupture test specimens of Modification IV and V alloys. For both of these alloys the fracture paths were associated with the large, eutectic colonies of primary gamma-prime. There was little evidence in these microstructures to rationalize the differences in mechanical properties between the two alloys. The improvement in mechanical properties between the Series III and II alloys, however, was clearly attributed to the microstructural differences evident in the respective failed test bar specimens. The photomicrographs of the failed Series II specimens shown in Figure 35 are characterized by fine grain sizes as well as the presence of continuous carbide networks throughout the microstructures. Comparison with the microstructure of the Series I TRW-NASA VIA failed test bar specimen shown in Figure 31a, on the other hand, suggests little difference between the structures of the Series III alloys, Figure 38. This in essence was reflected by the fact that little appreciable differences in times to reach 0.1% creep were observed between the Series I alloy and the Modification IV composition, Figure 37.

(d) Lubricant Compatibility Tests

Lubricant compatibility tests were conducted on the Series III metastable carbide alloys using OPT 112 and Deltaglaze 69 isothermal forging lubricants. Testing included 80 hours total exposure at each of three temperatures including 871°C (1600°F), 927°C (1700°F) and 982°C (1800°F). Evaluation included a metallographic examination of the oxidation/sulfidation attack and involved measurements of the depth of penetration of the reaction products. The results of these lubricant compatibility tests are shown in Table XXXII in terms of the depth of attack. The results indicated that for the Series III alloys the Deltaglaze 69 lubricant was somewhat more aggressive than the OPT 112 in its attack on the modified TRW-NASA VIA metastable carbide alloys. In general, the mode of attack of the forging lubricants involved a surface type corrosion rather than a localized penetration along grain boundaries or remnants of prior particle boundaries. Examples of the typical appearance of this generalized corrosion attack are shown in Figure 39 for the Modification IV and V alloys after 80 hours exposure at 982°C (1800°F). One feature apparent in the photomicrograph of the Modification IV alloy, Figure 39a, is the uneven nature of the attack along the surface. This effect was characteristic of the Modification IV composition at both 927°C (1700°F) and 982°C (1800°F). This unveven penetration was not associated with grain boundary areas or any other apparent discontinuities in the microstructure of the alloy. Although the depth of penetration was somewhat greater for the Modification IV alloy in terms of actual measurements, there appeared to be little significant difference between the two alloys in their resistance to attack by either of the forging lubricants. This reflected their general similarity in compositions.



(a) Modification IV, 100X



(b) Modification V, 100X

Figure 38. Light photomicrographs of Task III (metastable carbide alloys) Series II TRW-NASA VIA Modifications IV and V showing intergranular fracture mode typical of creep-rupture test specimens.

Table XXXII

Lubricant Compatibility Test Results for Task III (Metastable Carbide) Series III Alloys[1]

OPT 112 Lubricant	871°C (1600°F) mm	871°C (1600°F) inches	927°C (1700°F) mm	927°C (1700°F) inches	982°C (1800°F) mm	982°C (1800°F) inches
TRW-NASA VIA Mod IV	.00610	.00024	.01727	.00068	.02108	.00083
TRW-NASA VIA Mod V	.00229	.00009	.01397	.00055	.01905	.00075
Deltaglaze 69 Lubricant						
TRW-NASA VIA Mod IV	.00864	.00034	.02133	.00084	.02870	.00113
TRW-NASA VIA Mod V	.00584	.00023	.01651	.00065	.02362	.00093

(1) Measurements indicate depth of penetration after 80 hours exposure at the various test temperatures.

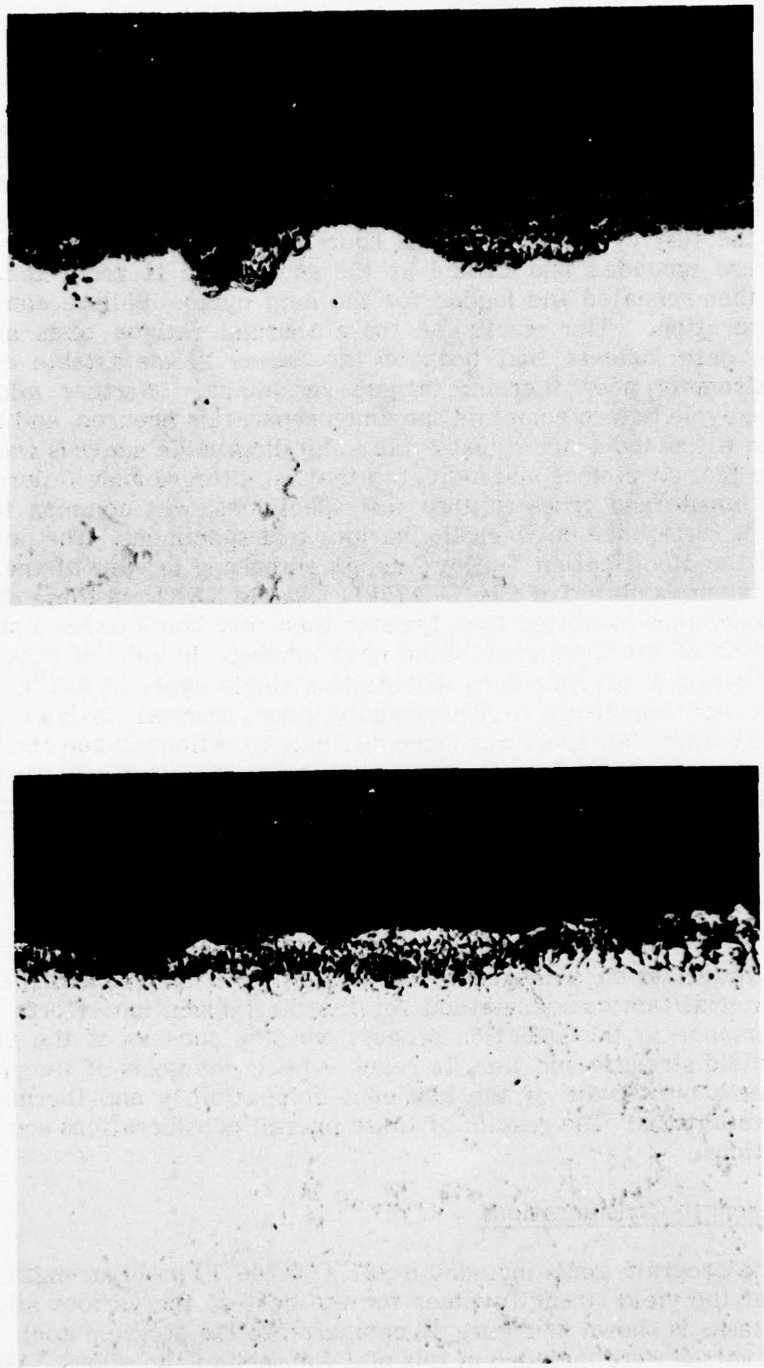


Figure 39. Light photomicrographs of Task III (metastable carbide) Series III alloys after 80 hours lubricant compatibility test with OPT 112 forging lubricant at 982°C (1800°F). Unetched condition. Magnification 500X.

(e) Thermal Fatigue/Creep Interaction Test Results

Thermal fatigue/creep interaction tests were conducted on two specimens from each of the Series III alloys. Processing for these specimens included HIP consolidation at 1316°C (2400°F)/103.4 MPa (15 ksi)/4 hours plus an aging treatment of 24 hours at 899°C (1650°F). The specimens were loaded at 927°C (1700°F)/172 MPa (25 ksi) and the test cycle ran for four hours under load and temperature after which specimens were unloaded and cooled at the same time to room temperature for 5-10 minutes and then reheated and loaded for the next cycle. Failure consisted of complete specimen separation. The results of these thermal fatigue tests are listed in Table XXXIII. The data indicate that both of the Series III metastable carbide superalloys exhibited extremely poor thermal fatigue resistance. Neither alloy exhibited lives exceeding one cycle before complete specimen separation occured, and the Modification V alloy failed to withstand a single test cycle. Metallographic analysis was conducted on the failed fatigue test specimens and indicated that an intergranular failure mode, similar to that for the tensile and creep-rupture test specimens, was common to both alloys, and there was little difference between the various test specimens. The poor thermal fatigue resistance of the Modification V alloy was not surprising in view of the rather poor creep rupture resistance exhibited at 954°C (1750°F), Table XXXI. In these creep-rupture tests, none of the specimens exhibited lives greater than four hours under a stress of 206.8 MPa (30 ksi), and one of the specimens failed upon loading. In view of this, the failure of any of the Modification V specimens to withstand a single cycle at 927°C (1700°F)/172 MPa (25 ksi) was not surprising. The equally poor thermal fatigue resistance of the Modification IV alloy, however, was more difficult to rationalize on the basis of the 954°C (1750°F) creep-rupture results. This particular alloy exhibited average rupture lives of 16 hours, and yet, none of the thermal fatigue specimens exceeded a single cycle at 927°C (1700°F)/172 MPa (25 ksi) under the thermal fatigue testing mode.

D. Task IV - Die Alloy/Process Selection

The efforts in this portion of the program included the analysis of the mechanical property characterization data generated in Tasks I, II and III and the selection of a single die material/fabrication method for the die fabrication efforts in Phase II. Of primary importance in the selection process was the success of the various alloys in meeting the yield strength and time to reach 0.1% creep goals of the program. Also of importance were the results of the lubricant compatibility and thermal fatigue/creep interaction evaluations. The results of these overall considerations are discussed in the following sections.

1. Strength Considerations

The program goals included a 927°C (1700°F) yield strength of 517 MPa (75 ksi). A plot of the yield strength values for the best of the various alloy compositions for all three tasks is shown in Figure 40 compared to the program goal. All of the yield strength data values were included in this plot for each of the alloys. All of these alloys were variations of the TRW-NASA VIA composition. The Task I (powder metallurgy) Modification II alloy featured a higher hafnium level compared to the base composition (2.29% versus 0.51%), tantalum reduced from 9.0% to 5.9% to balance the increase in

Table XXXIII

Thermal Fatigue/Creep Interaction Test Results for Task III (Metastable Carbide) Series III Alloys(1)

Alloy(2)	Cycles to Failure
TRW-NASA VIA	1
Modification IV	1
TRW-NASA VIA	0
Modification V	0

(1) Test Conditions: 25°C (70°F) - 927°C (1700°F), 0-172.4 MPa (25 Ksi), 4 hour cycle.

(2) Processing conditions for all specimens included HIP at 1316°C (2400°F)/103 MPa (15 Ksi)/4 hours plus 899°C (1650°F)/24 hours/air cool heat treatment.

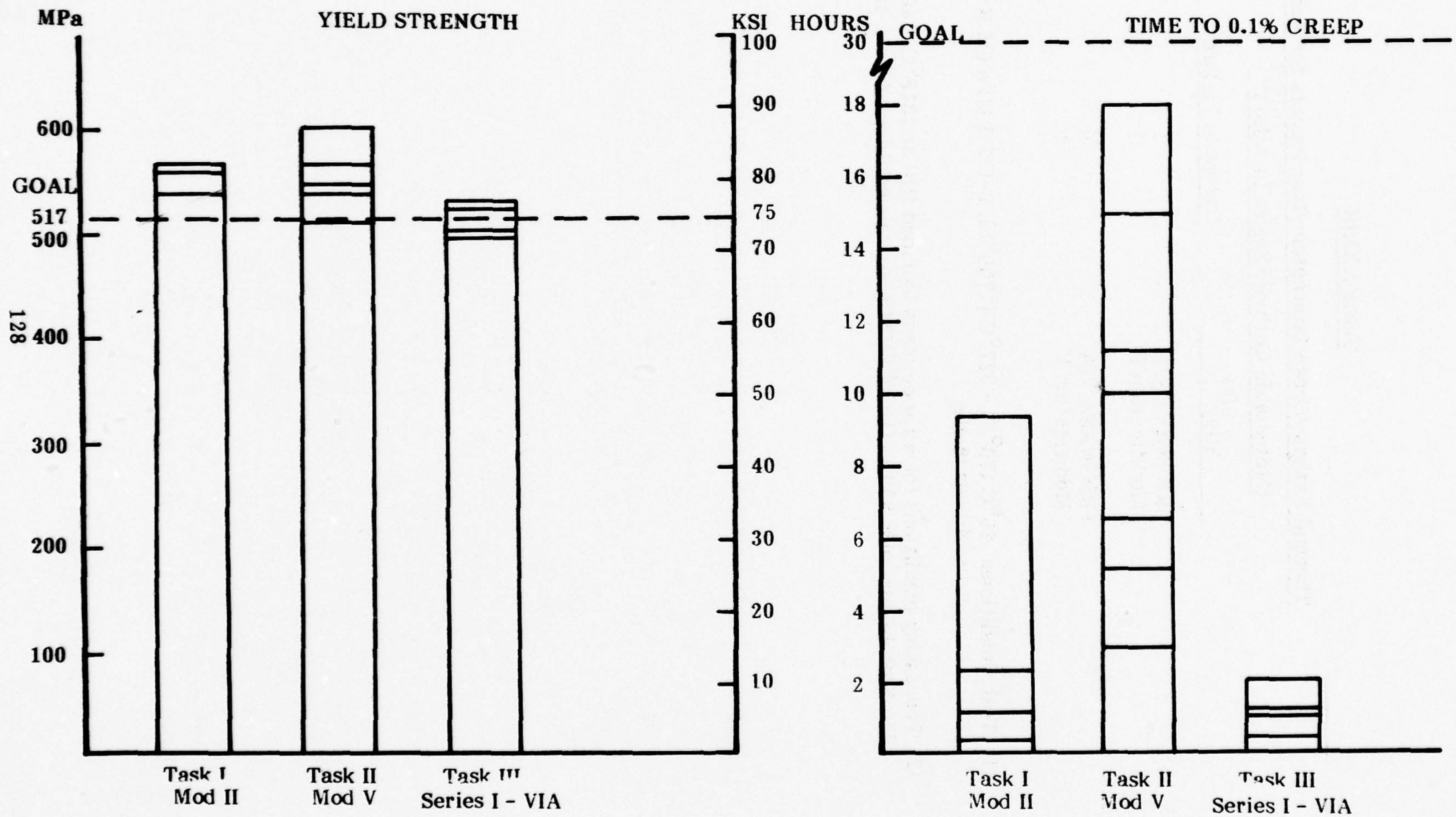


Figure 40. Yield strength and time to 0.1% creep for the best of the Task I (powder metallurgy) Task II (casting superalloy) and Task IV (metastable carbide)alloys.

gamma-prime content anticipated by the hafnium increase and had rhenium added at the 0.23% level. The Task II (casting superalloy) Modification V alloy was basically quite similar in composition to the powder metallurgy alloy. In the casting alloy, however, the hafnium content and rhenium contents were somewhat lower than in the powder metallurgy alloy. The hafnium was at the 1.78% level compared to 2.29% for the powder metallurgy alloy and the rhenium was at 0.18% compared to 0.23%. The Task III (metastable carbide alloy) was the Series I base TRW-NASA VIA composition. This composition differed from the original casting alloy developed under NASA sponsorship (29) in that rhenium was removed from the alloy and carbon was added in the form of metastable SiC particles during the attrition of the base alloy powders.

Analysis of the tensile strength results indicated that, in general, the values were comparable for the three alloys. As shown in terms of yield strength, Figure 40, the three alloys all showed potential for meeting the program goal of 517 MPa (75 ksi). On the average, all of the alloys did meet or exceed the program goal. The powder metallurgy composition exhibited an average yield strength of 574.4 MPa (83.3 ksi) compared to the 551.6 MPa (80.0 ksi) average for the casting alloy and the 517 MPa (75 ksi) average for the metastable carbide alloy. There was more scatter evident in the casting alloy results, but this was attributed to the fact that certain specimens were heat treated and certain specimens were machined from different locations within the cast blocks. A section size effect was thus encountered. In terms of ultimate tensile strength, there was no difference between the various compositions. The powder metallurgy alloy exhibited the highest average ultimate tensile strength at 663.3 MPa (96.2 ksi) while the casting alloy exhibited the lowest average at 655 MPa (95.0 ksi). The ductility values for these alloys were also similar, with the single exception of the percent reduction area for the casting alloy. The average percent elongation values ranged from 2.9% (powder metallurgy alloy) to 3.8% (casting alloy). At an average value of 5.0%, however, the percent reduction of area for the casting alloy was double that exhibited by the powder metallurgy and metastabale carbide alloys, both exhibiting an average value of 2.5%. In summary then, the tensile test results for the various alloys at 927°C (1700°F) suggested that all three alloy/processing combinations exhibited sufficient potential for application as isothermal forging die materials.

2. Creep-Rupture Considerations

The program goals included a time to reach 0.1% creep of 30 hours at test conditions of 954°C (1750°F)/206.8 MPa (30 ksi). A plot of the creep-rupture results in terms of the various values of time to reach 0.1% creep for the best of the alloy compositions for all three tasks is also shown in Figure 40 compared to the program goal. All of the creep-rupture results were included in this plot for each of the alloys. The alloy compositions included in this plot are identical to those of the yield strength plot since the alloys exhibited superior properties in terms of both yield strength and times to reach 0.1% creep. In marked contrast to the tensile results, the creep-rupture results indicated a considerable difference between the metastable carbide and powder metallurgy alloys and the casting composition. This was manifested primarily in terms of time to reach 0.1% creep and in rupture life. For the casting superalloy, times to reach 0.1% creep ranged from 3.2 to 18.2 hours, while the values for the powder metallurgy alloy ranged from 0.37-9.5 hours. The metastable carbide alloy exhibited extremely poor creep-rupture properties with times to reach 0.1% creep ranging from

0.45 to 2.02 hours. While the 18.2 hour maximum value exhibited by a cast specimen was below the desired program goal of 30 hours, it did represent a considerable improvement in the properties of IN-100 (the currently used nickel-base superalloy isothermal forging die material) cast in similar thick sections, Table III. The differences in creep-rupture resistance were even more apparent when rupture lives were considered. The casting alloy exhibited an average rupture life of 120.0 hours, compared to 19.8 hour average of the powder metallurgy specimens and the 25.6 hour average of the metastable carbide alloys. In contrast to the differences in time to reach 0.1% creep and the rupture lives, the creep ductility properties were comparable for the three alloys. The percent elongation ranged from 1.4% (powder metallurgy alloy) to 3.1% (casting alloy) and the reduction of area ranged from 0.6% (powder metallurgy alloy) to 1.9% (both the casting and the metastable carbide alloys). It was apparent that in spite of the large equiaxed grain size achieved in both the powder metallurgy and metastable carbide alloys, the creep-rupture properties (rupture life and time to reach 0.1% creep) were considerably inferior to those of the casting alloy. While properties exceeding those of cast material have been achieved in powder materials featuring columnar grained structures, (e.g., RSR alloys (23) and oxide dispersion strengthened alloys (60)), the load distributions experienced in isothermal forging die applications indicate little advantage to be gained by employing die materials featuring the columnar grained types of structures (61). In summary, then, the creep-rupture test results suggested that only the casting alloy exhibited sufficient potential for application as an isothermal forging die material.

3. Lubricant Compatibility Considerations

Lubricant compatibility tests were conducted only on the Series III alloys from each task and included exposure of the various alloys to OPT 112 and Deltaglaze 69 isothermal forging lubricants over a range of temperatures. Since the creep-rupture evaluations indicated that only the cast superalloys exhibited sufficient potential for isothermal forging die applications, only the lubricant compatibility results for the casting alloys were considered in the die alloy/process selection evaluation. The results of these lubricant compatibility tests for the Modification IV and V alloys are listed in Table XXI. As a comparison with the currently used superalloy isothermal forging die, tests were also run on IN-100 material from the Series I study and these results are also listed in Table XXI. Although the depth of penetration was somewhat greater for the Modification V alloy in terms of actual numbers, there appeared to be little significant difference between the two alloys in their resistance to attack by the forging lubricants. This reflected the fact that the two alloys were generally similar in composition. There was, however, a significant difference in resistance to attack by the forging lubricants in comparison to the IN-100 alloy. The resistance to attack of the modified TRW-NASA VIA casting alloys was superior to that of IN-100 at all three temperatures. The mode of attack on the IN-100 material was similar to that of the Series III alloys and was characterized by a generalized surface type corrosion as opposed to an intergranular type attack. In summary, the lubricant compatibility test results indicated that both of the Series III casting modifications of TRW-NASA VIA exhibited comparable resistance to attack by forging lubricants and further that they were superior in resistance compared to the currently used IN-100 forging die material.

4. Thermal Fatigue/Creep Interaction Considerations

Similar to the lubricant compatibility testing, thermal fatigue/creep interaction tests were conducted only on Series III alloys. Since only the cast superalloys exhibited sufficient potential for isothermal forging die applications, only the thermal fatigue results for the casting alloys were considered in the die alloy/process selection evaluation. In fact, as the thermal fatigue data presented in Tables XI, XXII and XXXIII indicate, the casting superalloys were clearly superior in thermal fatigue resistance compared to the powder metallurgy and the metastable carbide alloys. The results of the thermal fatigue tests on the casting superalloys are listed in Table XXII and indicate that Modification IV alloy was superior to Modification V alloy in thermal fatigue/creep interaction resistance in both the as-cast condition and after the 899°C (1650°F) age for 24 hours. In the as-cast condition the fatigue life of Modification IV was triple that of Modification V, while that of the aged material was double that of aged Modification V. Metallographic analysis of failed test bars indicated that there was little difference between the various specimens to account for the variations observed in fatigue response. The thermal fatigue results thus suggested that the Modification IV alloy, containing the higher hafnium content (2.1% versus 1.78%) and the higher tantalum content (8.79% versus 6.79%) exhibited stronger potential for isothermal forging die applications.

5. Summary

Consideration of the mechanical property test results indicated that in spite of the high ultimate tensile strengths and yield strengths exhibited by the powder metallurgy and metastable carbide alloys, the casting superalloys overall indicated a much greater potential for isothermal forging die applications. The creep-rupture results (both time to reach 0.1% creep and rupture life) and the thermal fatigue results for the casting alloys were clearly superior to those of the powder metallurgy and metastable carbide alloys. Lubricant compatibility testing indicated that the casting alloys offered superior resistance to IN-100, currently in use as an isothermal forging die material. The primary question then resolved itself as to the selection of a recommended casting composition for the die fabriction efforts of Phase II. Both casting alloy Modification IV and V indicated strong potential for use as isothermal forging die materials. As mentioned previously in the discussion concerning the thermal fatigue results, the basic difference between the two alloys was that Modification IV contained higher hafnium and tantalum compared to the Modification V alloy.

In terms of strength at 927°C (1700°F), both alloys essentially exhibited comparable properties, with average ultimate tensile strengths of approximately 655.0 MPa (95 ksi) and an average yield strength of 540.7 MPa (78.4 ksi) for Modification IV and 553.7 MPa (80.3 ksi) for Modification V. The creep-rupture properties at 954°C (1750°F) were also comparable, with Modification IV exhibiting a maximum rupture life of 171.1 hours compared to the 159.3 hour maximum for Modification V. In terms of time to reach 0.1% creep, however, Modification V exhibited a maximum of 18.2 hours compared to the 14.4 hours of Modification IV. Both of the alloys exhibited comparable resistance to exposure to the isothermal forging lubricants at elevated temperature. The only significant difference observed in the mechanical properties was in the

thermal fatigue/creep interaction results, where Modification IV alloy was clearly superior to the Modification V composition. It must be pointed out, however, that only single tests were conducted at the various conditions. In view of these considerations a compromise was reached in terms of a recommended chemistry for the casting alloy. On the basis of the thermal fatigue results, the chemistry range selected basically included the Modification IV alloy, with one important exception. Whereas the Modification IV alloy featured tantalum at approximately 9.0%, the alloy selected for the forging dies specified a range of 6.0-7.0% for tantalum. This decision was reached partly on the basis of the currently escalating costs for tantalum (62). The chemistry specification for the casting superalloy selected for the Phase II die fabrication efforts is listed in Table XXXIV.

Table XXXIV

Modified TRW-NASA VIA Casting Superalloy Chemistry Range in Weight Percent Selected for Phase II Die Fabrication

Element	Chemistry Range
C	.03-.05
Al	5.40-5.80
B	.006-.010
Cb	.04-.60
Co	7.40-7.69
Cr	5.80-6.20
Hf	1.80-2.10
Mo	1.90-2.10
Ta	6.0-7.0
Ti	0.9-1.0
W	5.5-6.0
Zr	.01-6.0
Re	.20-.22
O_2	25 ppm Maximum
N_2	25 ppm Maximum

IV - PHASE II - FORGING DIE FABRICATION

A. Selection of Structural Component

The selection of a demonstration structural component for evaluation on this program was coordinated with Air Force Contract F33615-76-C-5386, "Isothermal Forging of Beta Titanium". Requirements for the beta titanium program necessitated the selection of two components, a fatigue critical part and a static critical part in the size range up to 1290 square cm (200 square inches) plan area. Selection of the component was also coordinated with McDonnell Aircraft Company (MCAIR) whose F-15 fighter currently is the most productive advanced aircraft and uses a high volume fraction of titanium alloy components. In a typical fighter aircraft at MCAIR there are more than five hundred titanium alloy parts in the size range up to 1290 square cm (200 square inches) plan area.

For this program a static critical component was selected as shown in the drawing in Figure 41 and the photograph in Figure 42. The part is referred to as a trunnion and is currently machined from a conventional oversize die forging procured in annealed Ti 6Al-6V-2Sn. The part contains extensive thin web areas, reinforcing ribs, and a slightly heavier section for the arresting gear drag brace attachment. Functionally, it is primarily required to transfer high loads from the arresting gear drag brace into the aft fuselage structure through fasteners around the periphery of the part. If produced as an isothermal forging, extensive machining time could be saved if net rib and web areas are provided during forging. In addition, forging of the trunnion is a challenge because of the large differences in metal distribution in the part and the large, deep rib required for the arresting gear drag brace attachment. The large differences in metal distribution will cause non-uniform die loading during forging which will provide maximum stress conditions on the dies, a undesirable condition which occurs often in practice and will provide a test for the dies fabricated in this program.

The finish machined trunnion component webs vary in thickenss from 0.15 cm-0.20 cm (0.06 to 0.08-inch) while the rib width varies from 0.20-0.30 cm (0.08 to 0.12-inch). The maximum rib depth is 1.17 cm (0.46-inches) except where the attachment is made. The depth of the rib in this area is 4.3 cm (1.7-inches). The average overall part dimensions are approximately 30 cm (12-inches) wide by 26.7 cm (10.5-inches) long with a total plan area of about 800 square cm (124 square inches).

In designing the part as an isothermal forging several changes were made which are shown in Figure 43. The part followed the machined part outline with some flash extension and a .06-cm (0.02-inch) chem-mill allowance per surface. The chem-mill allowance was added for two reasons; first to insure removal of any alpha case that may arise during forging and second, it is not practical to forge webs less than 0.3 cm (0.1 inch) in thickness for the size range of the component. The small ribs were allowed to maintain their natural draft, however, a 7^{o} draft angle was necessary to achieve release of the part after forging. Machining in this area will be required after forging. The forging design was reviewed by MCAIR engineers and found to be acceptable and, in addition, cost effective with respect to the reduced machining requirements after forging.

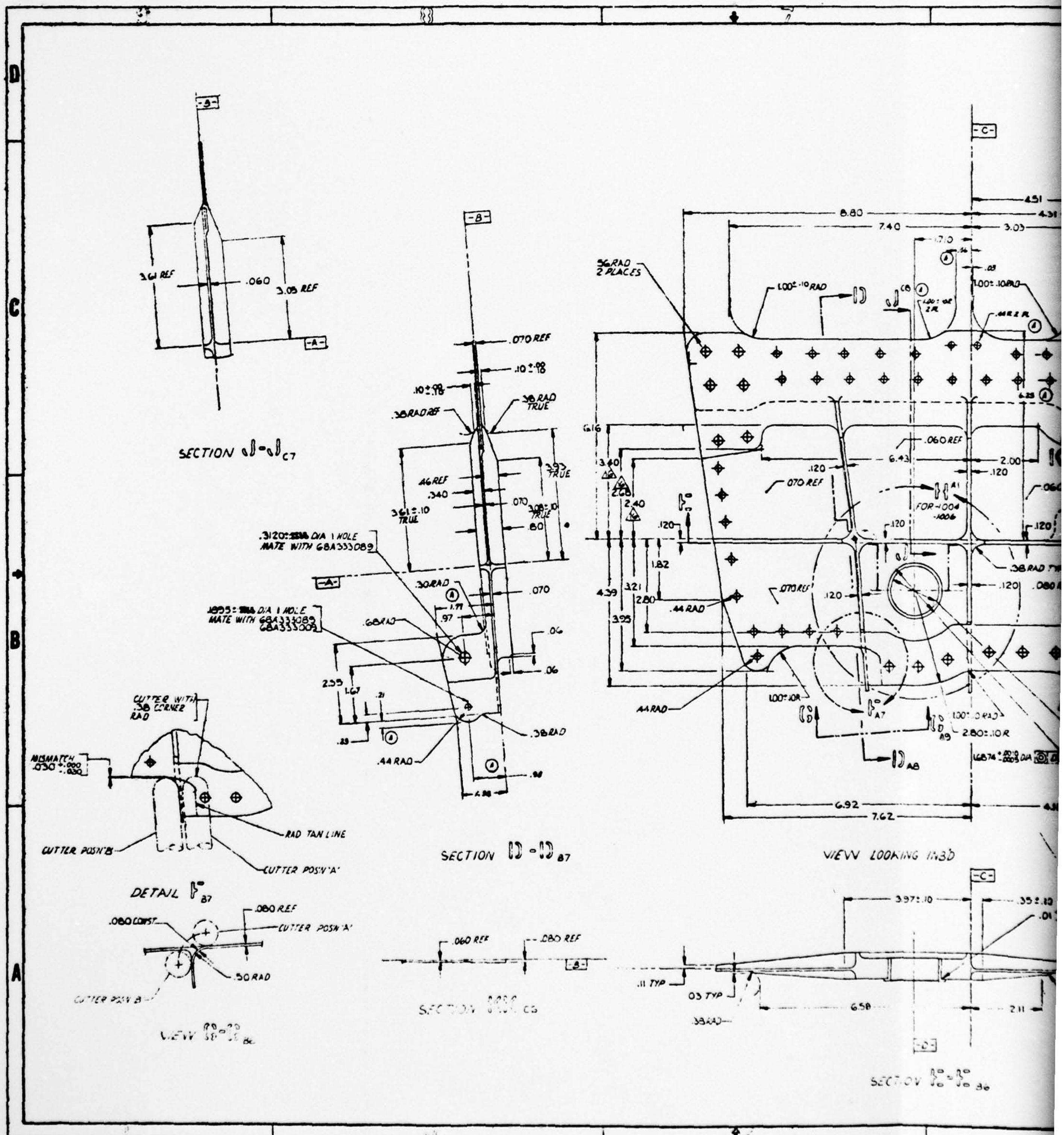
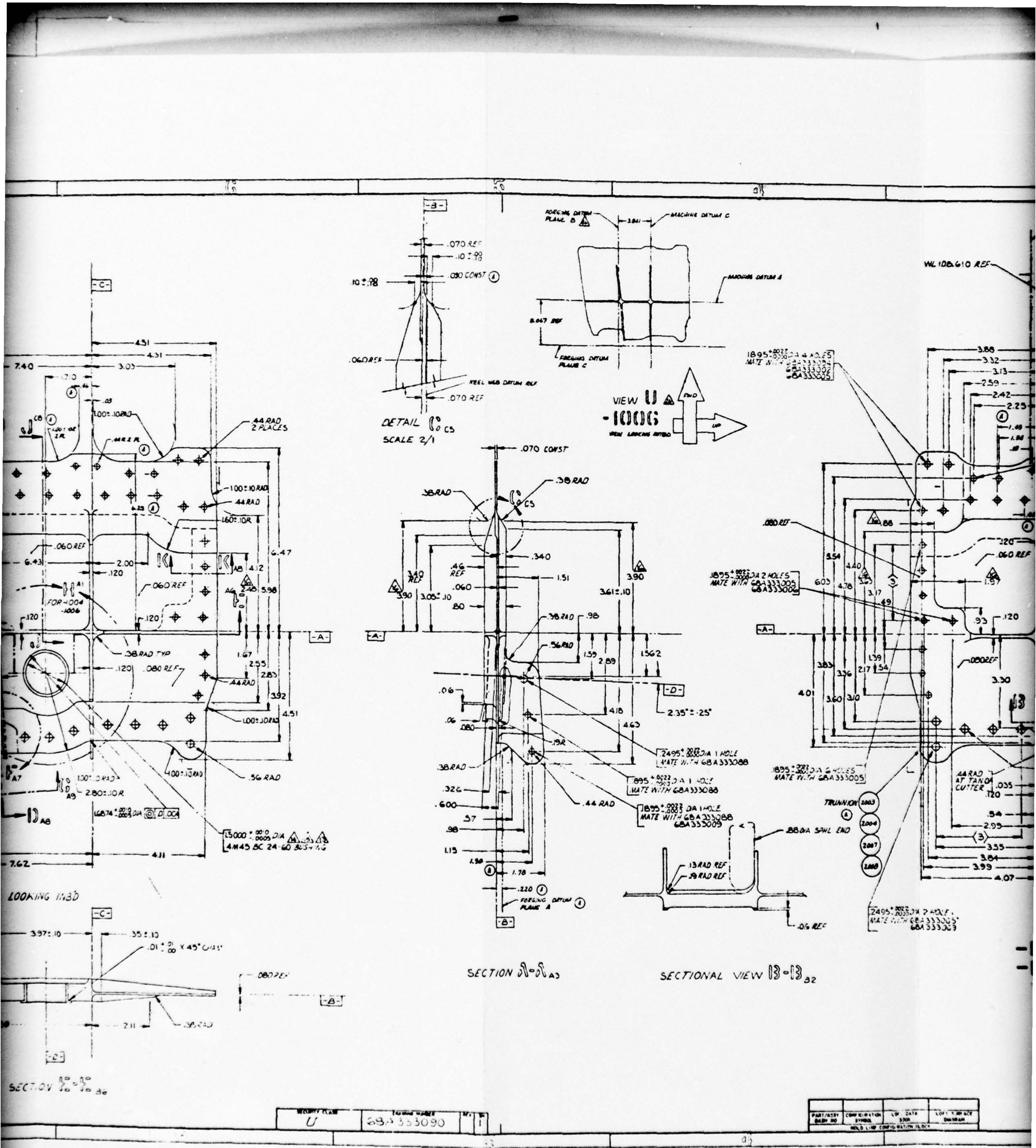
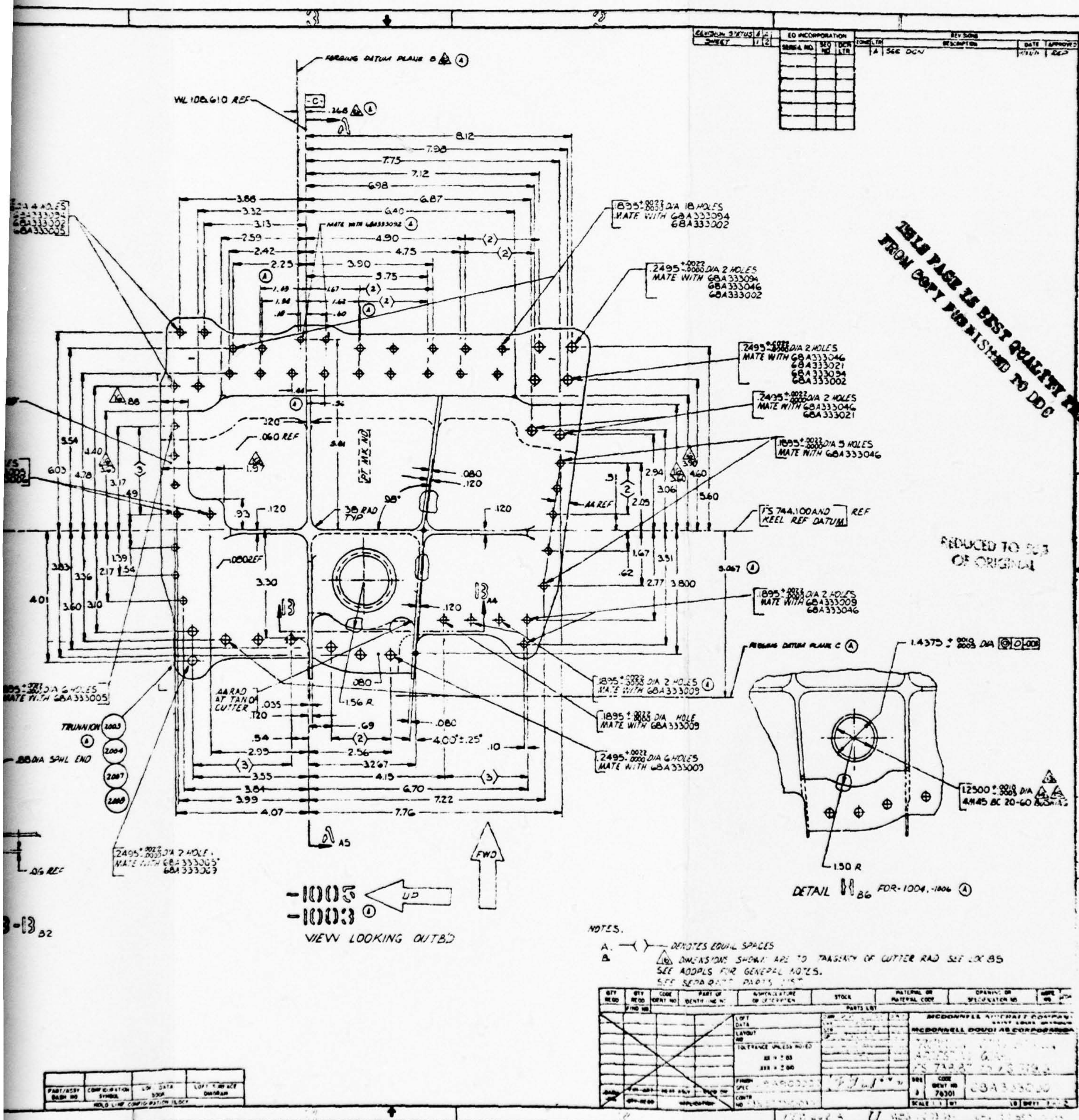


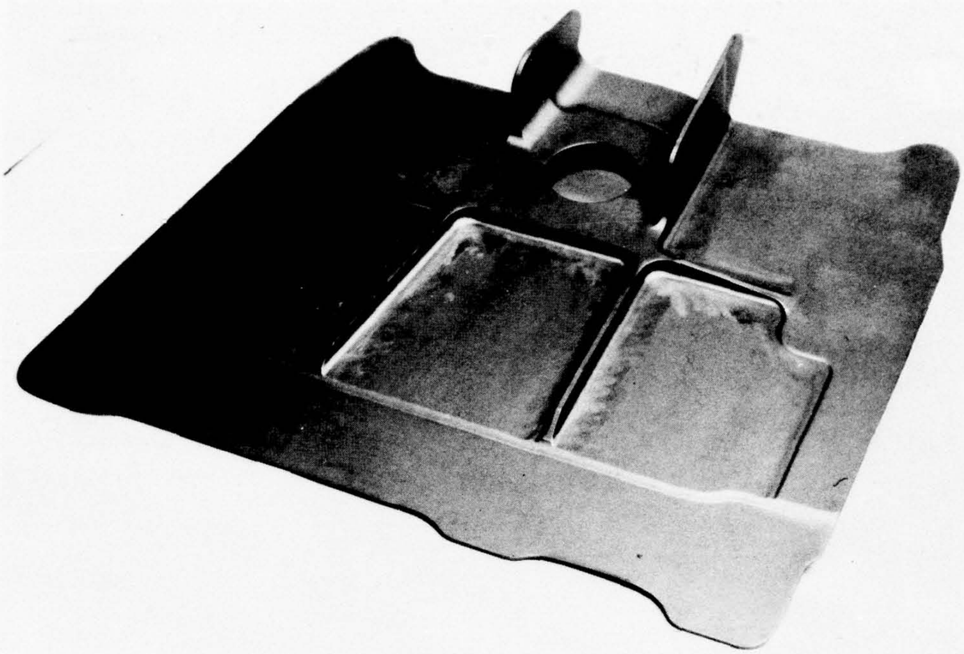
Figure 41. Drawing for trunnion drag brace arresting gear (P/N 68A333090).

DETAIL C CS
SCALE 2/1
VIEW U
-1006
SECTION A-A A3
SECTIONAL VIEW B-B B2
SECTION E-E
LOOKING INBD
FORGING DATUM PLANE B
MACHINE DATUM C
FORGING DATUM PLANE C
FORGING DATUM PLANE A
KEEL WEB DATUM REF
.070 CONST
.88 DIA SPHL END
.13 RAD REF
.06 REF
WL 108.610 REF
TRUNNION
.2495 DIA 1 HOLE MATE WITH 68A333088
.1895 DIA 1 HOLE MATE WITH 68A333088
.1895 DIA 1 HOLE MATE WITH 68A333088 68A333009
.1895 DIA 2 HOLES MATE WITH 68A333005 68A333006
.1895 DIA 6 HOLES MATE WITH 68A333005
.2495 DIA 2 HOLES MATE WITH 68A333005 68A333009
.1895 DIA 4 HOLES MATE WITH 68A333004 68A333002 68A333025
.5000 DIA 4M45 BC 24-60 BUSHING
FOR -1004 -1006
.44 RAD 2 PLACES
.38 RAD TYP
.56 RAD
.44 RAD AT TAN OF CUTTER
SECURITY CLASS
U
DRAWING NUMBER
68A333090
PART/ASSY DASH NO
CONFIGURATION SYMBOL
LOF DATA
LOFT SURFACE DIAGRAM
WELD LINE CONFIGURATION BLOCK

B

2


FORGING DATUM PLANE B
WL 108.610 REF
.1895 +.0022 -.0000 DIA 18 HOLES MATE WITH 68A333094 68A333002
.2495 +.0022 -.0000 DIA 2 HOLES MATE WITH 68A333094 68A333046 68A333002
.2495 +.0022 -.0000 DIA 2 HOLES MATE WITH 68A333046 68A333021 68A333094 68A333002
.2495 +.0022 -.0000 DIA 2 HOLES MATE WITH 68A333046 68A333021
.1895 +.0022 -.0000 DIA 5 HOLES MATE WITH 68A333046
FS 744.100 AND KEEL REF DATUM REF
REDUCED TO 50% OF ORIGINAL
.1895 +.0022 -.0000 DIA 2 HOLES MATE WITH 68A333009 68A333046
FORGING DATUM PLANE C
1.4375 +.0010 -.0005 DIA
.1895 +.0022 -.0000 DIA 2 HOLES MATE WITH 68A333009
.1895 +.0022 -.0000 DIA HOLE MATE WITH 68A333009
.2495 +.0022 -.0000 DIA 6 HOLES MATE WITH 68A333009
1.2500 +.0010 -.0005 DIA 4.445 BC 20-60 BUSHING
1.50 R
DETAIL H B6 FOR -1004, -1006
TRUNNION
.88 DIA SPHL END
AA RAD AT TAN OF CUTTER
1.56 R
4.00° ±.25°
FWD
UP
-1005
-1003
VIEW LOOKING OUTB'D
B-B B2
NOTES.
A. —( )— DENOTES EQUAL SPACES
B. DIMENSIONS SHOWN ARE TO TANGENCY OF CUTTER RAD SEE LOC B5
SEE ADDPLS FOR GENERAL NOTES.
SEE SEPARATE PARTS LIST
REVISION STATUS SHEET
EO INCORPORATION
REVISIONS
A SEE DCN
MCDONNELL AIRCRAFT COMPANY
MCDONNELL DOUGLAS CORPORATION
76301
68A333000
HOLD LINE CONFIGURATION BLOCK



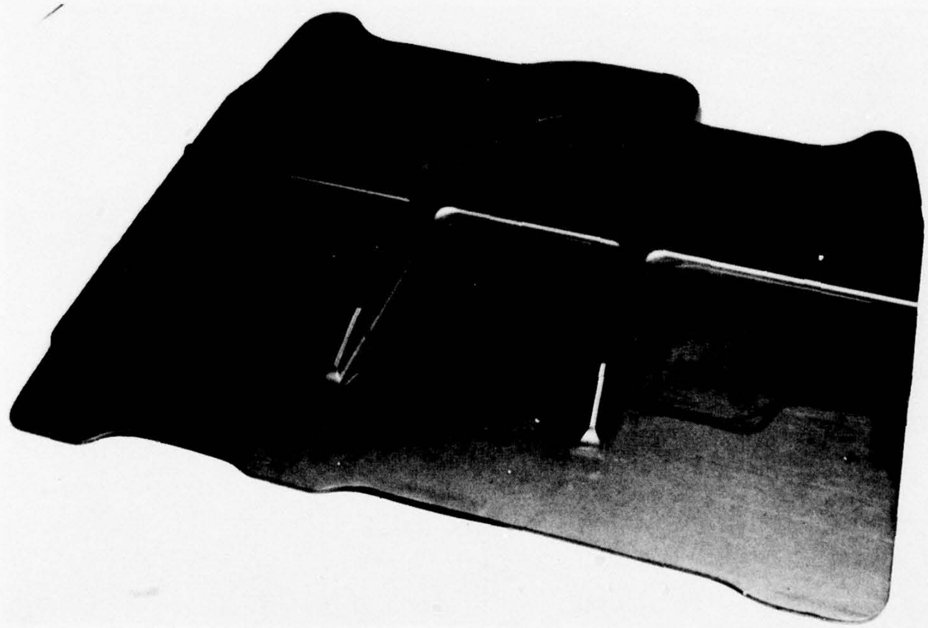
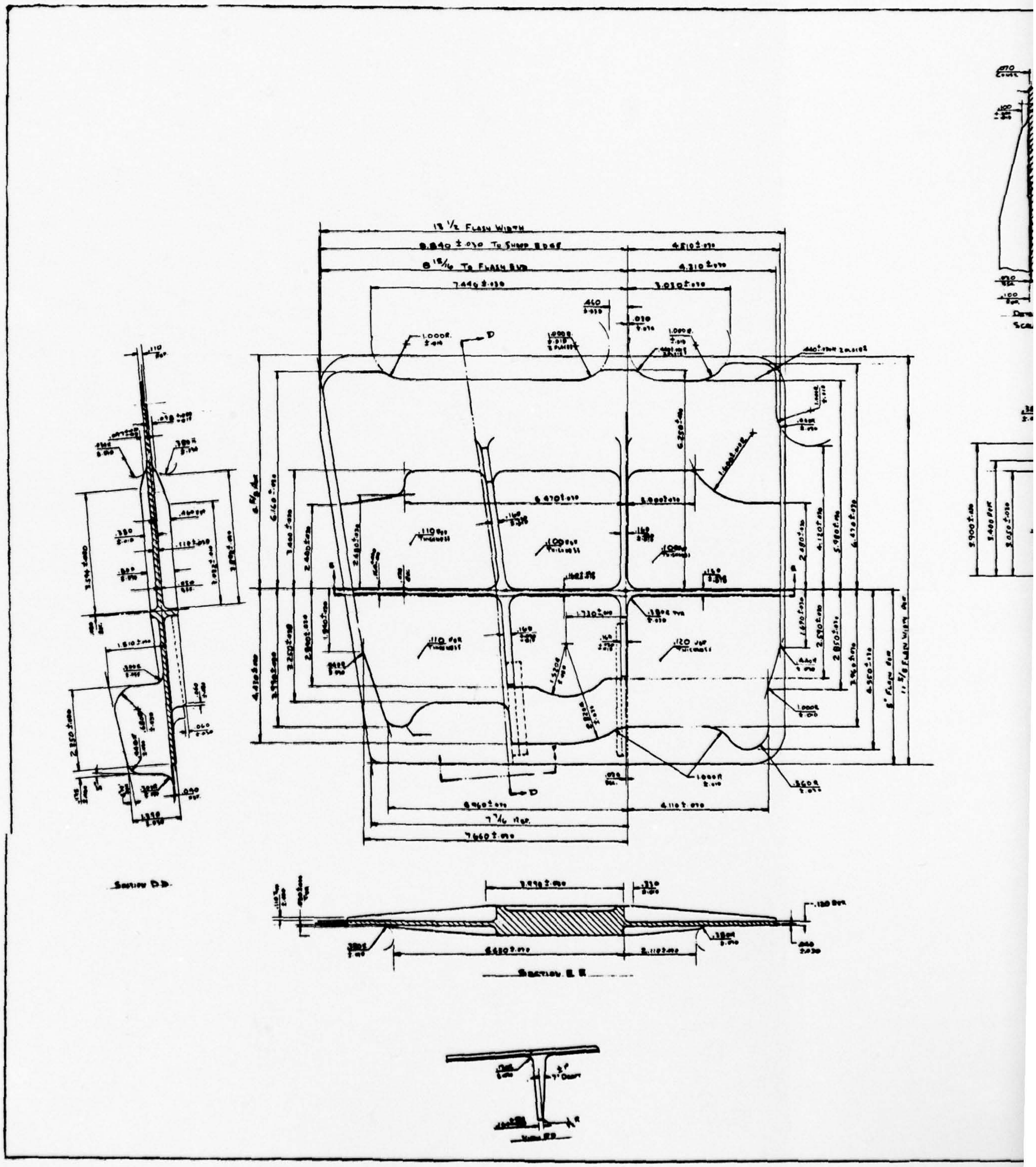
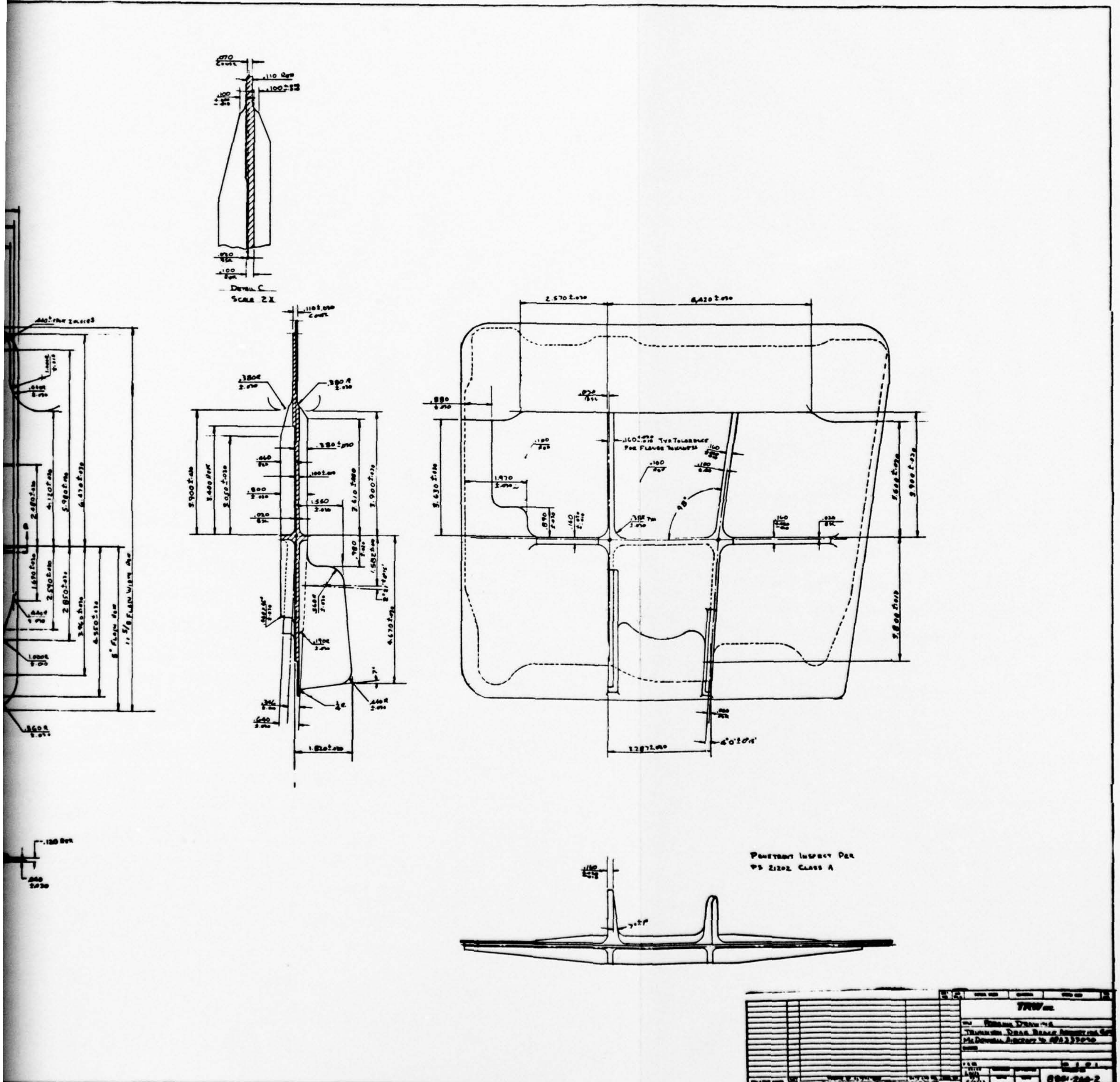


Figure 42. Final machined trunnion drag brace arresting gear component.



A

Figure 43. Forging drawing for trunnion dr



drawing for trunnion drag brace arresting gear (P/N 68A333090).

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2

B. Design of Hot Die Tooling and Support System

The hot die forging system was built using a modular tooling design concept. In isothermal forging the total cost of the die system often dictates the cost effectiveness of the process. Because the number of components produced for aircraft structural application is usually of low volume (several hundred pieces per year) the tooling costs can significantly add to the total cost of the production part and the break even point compared to some other manufacturing (conventional forging or machining) may be several years into the aircraft build program. The modular tool concept allows the use of a support system for several production components while for each part only having to replace the die and punch. The die and punch can be considered as die inserts. In order to use this concept the tooling system has to be designed for a "family" of parts, that is the plan area should be in a defined range and the parts should be of a similar plan view shape. For example, the trunnion is of a square plan view with a planar area of about 800 square cm (124 square inches). Parts approaching this size and shape could be produced using this die system; however, a part with a 5 cm (2 inch) width and 76.2 cm (30 inch) length, although smaller in plan area, could not be produced in this die system because of its long rectangular shape. Some of the pertinent features of the system are discussed below.

A bill of materials for the tooling system is given in Table XXXV. A schematic representation for the system is shown in Figure 44. The major components included support plates, stack plates, heater plates, a die holder which also acted as a heater and the die inserts. The top and bottom support plates were machined from mild steel. These are shown in Figure 45. The design of the plates allowed for mechanical attachment of the remaining assembly (stack plates, heater plates, holder and inserts). Located below the bottom support plate was an ejection system (see Figure 46) attached mechanically to the stainless steel stack plate through four holes drilled through the support plate. An independent ejection system was used to obtain latitude in the selection of a hydraulic press. Not all presses are equipped with ejection systems, and the ejection assembly shown was built specifically for use with hot die tooling. The ejection system was used to raise and lower the bottom die insert. Directly attached to the upper support plate were mild steel spacers and two ejection cylinders. The cylinder was mechanically attached to ejection pins that were located in holes drilled through the stack plate, insulation, heater plate and die insert. These pins were used for ejection of the parts after forging. Part of the upper tooling system is shown in Figure 47 illustrating the location of the ejection mechanism. The deep ribbed areas of the part were designed into the upper die insert, and based on experience, the part is expected to remain in the upper die (thus the need for ejection).

As noted in Figure 44, stainless steel spacer blocks were used followed by insulation material between the spacer and heater blocks. The insulation material was of laminated construction 0.15-cm (0.06-inch) stainless steel and ceramic paper. The laminated construction has been found to maintain dimensional stability after initial use and provide insulation between the stack and heater plates. The heater plates were cast from IN-100 material one of which is shown in Figure 48. All heating was conducted using resistance heaters (or cartridge heater rods). The bottom heater plate used eight rods with 9.8-cm (3 7/8-inch) heated length located vertically in the plate. The upper heater plate utilized seven rods also 9.8-cm (3 7/8-inch) heated length located vertically in the plate. Attached

Table XXXV

Bill of Materials for Hot Die Tooling and Support System

Major Component	Quantity	Material
Bottom Support Base	1	AISI 1020
Top Support Base	1	AISI 1020
Bottom Spacer Plate	1	310 SS
Top Spacer Plate	1	310 SS
Bottom Heater Plate	1	IN-100
Top Heater Plate	1	IN-100
Die Ring (Holder)	1	IN-100
Riser Blocks	4	AISI 1020
Ejection Pins - Top	3	IN-100
Ejection Cylinder - Bottom	1	TRW Built
Hydraulic Cylinders - Top	2	Jergens
Top Ejector Plates	3	AISI 1020
Resistance Heaters	19	Watlow

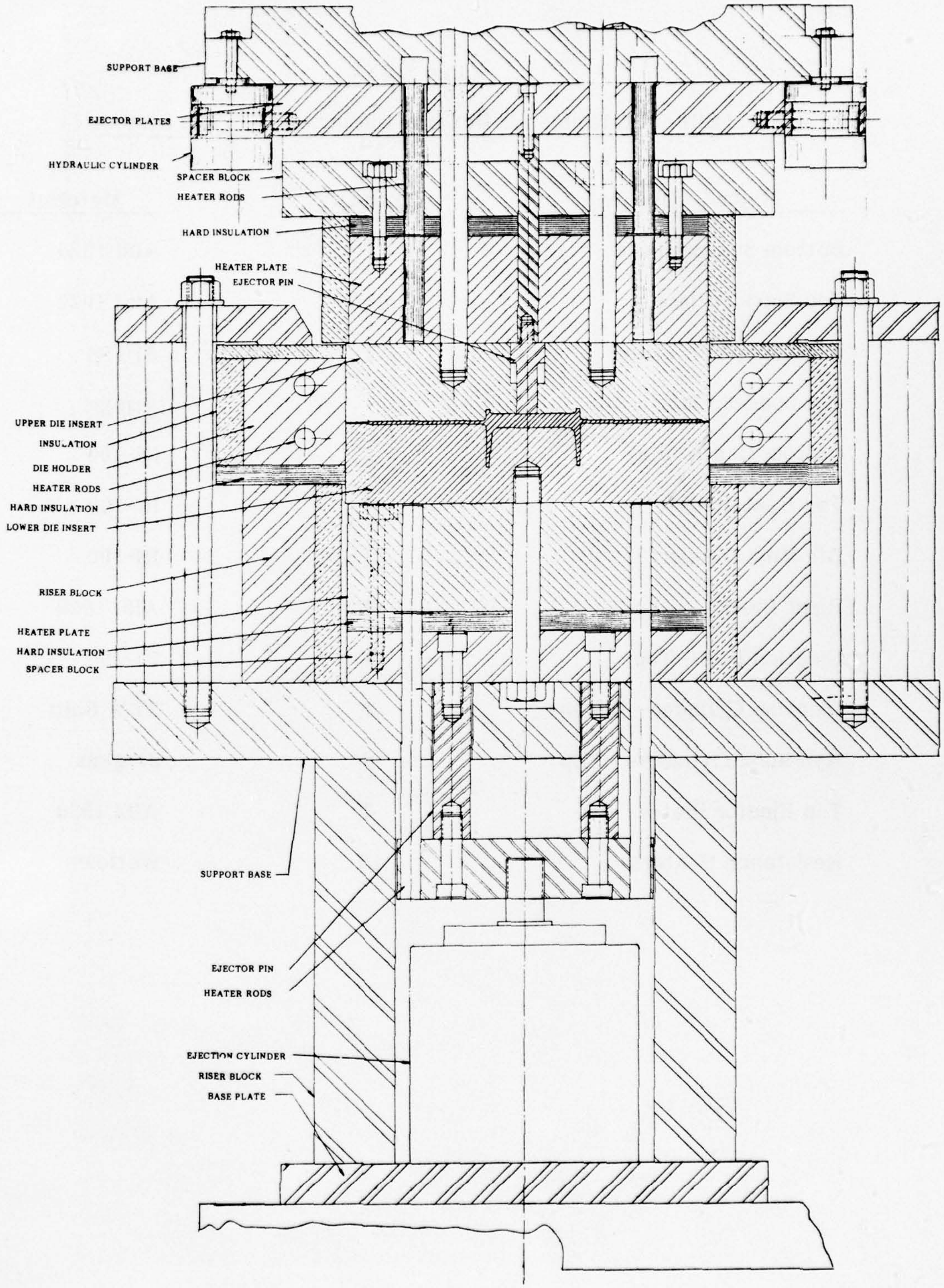
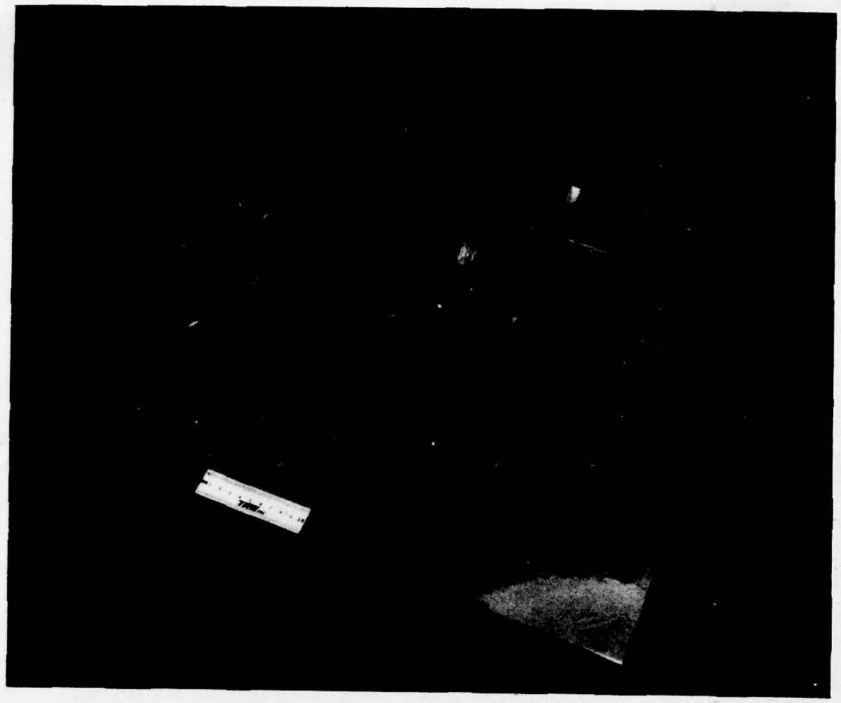


Figure 44. Schematic representation of tooling system.



a. Upper support plate

b. Lower support plate

Figure 45. Support plates.

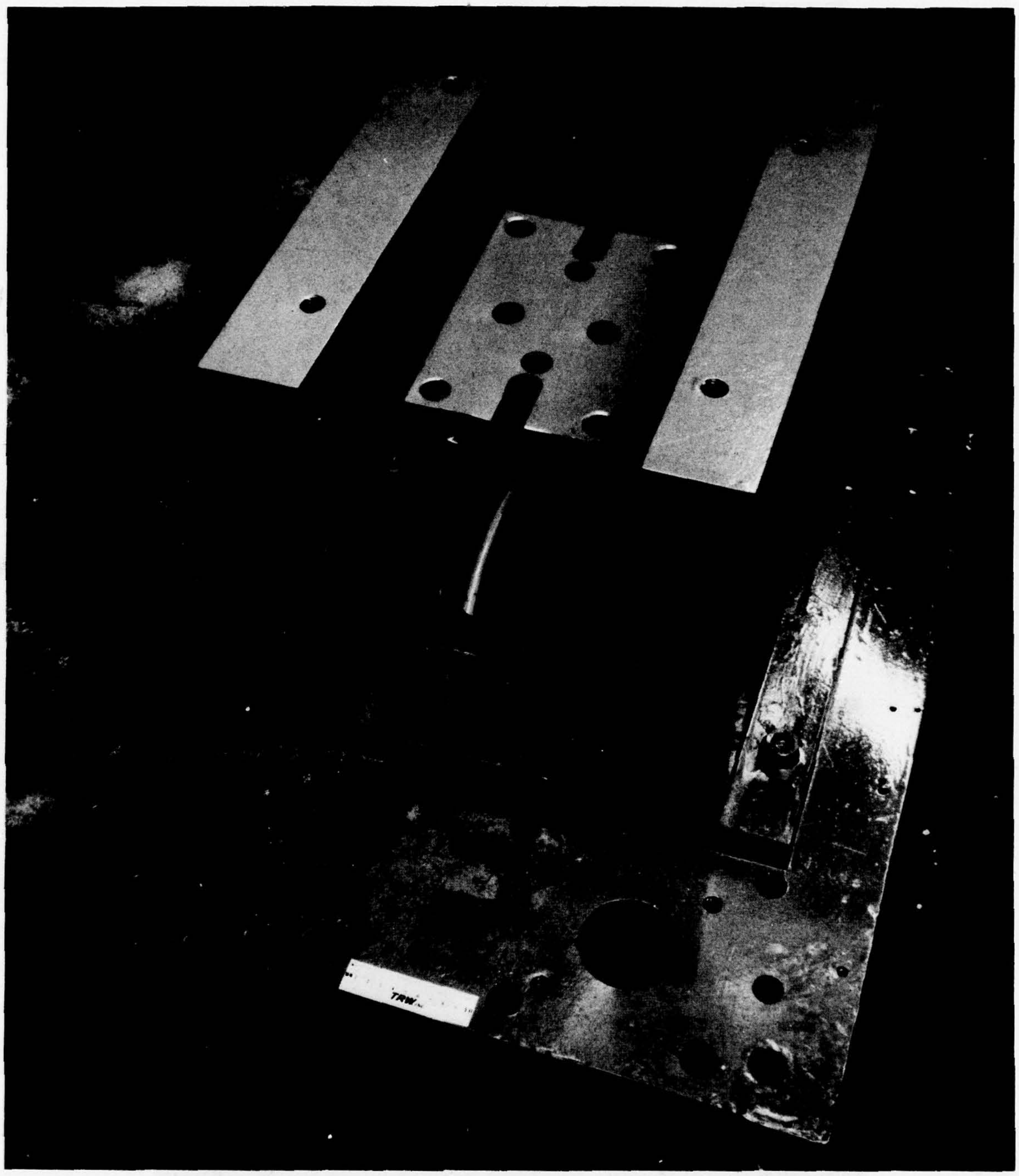


Figure 46. Lower die insert ejection system.

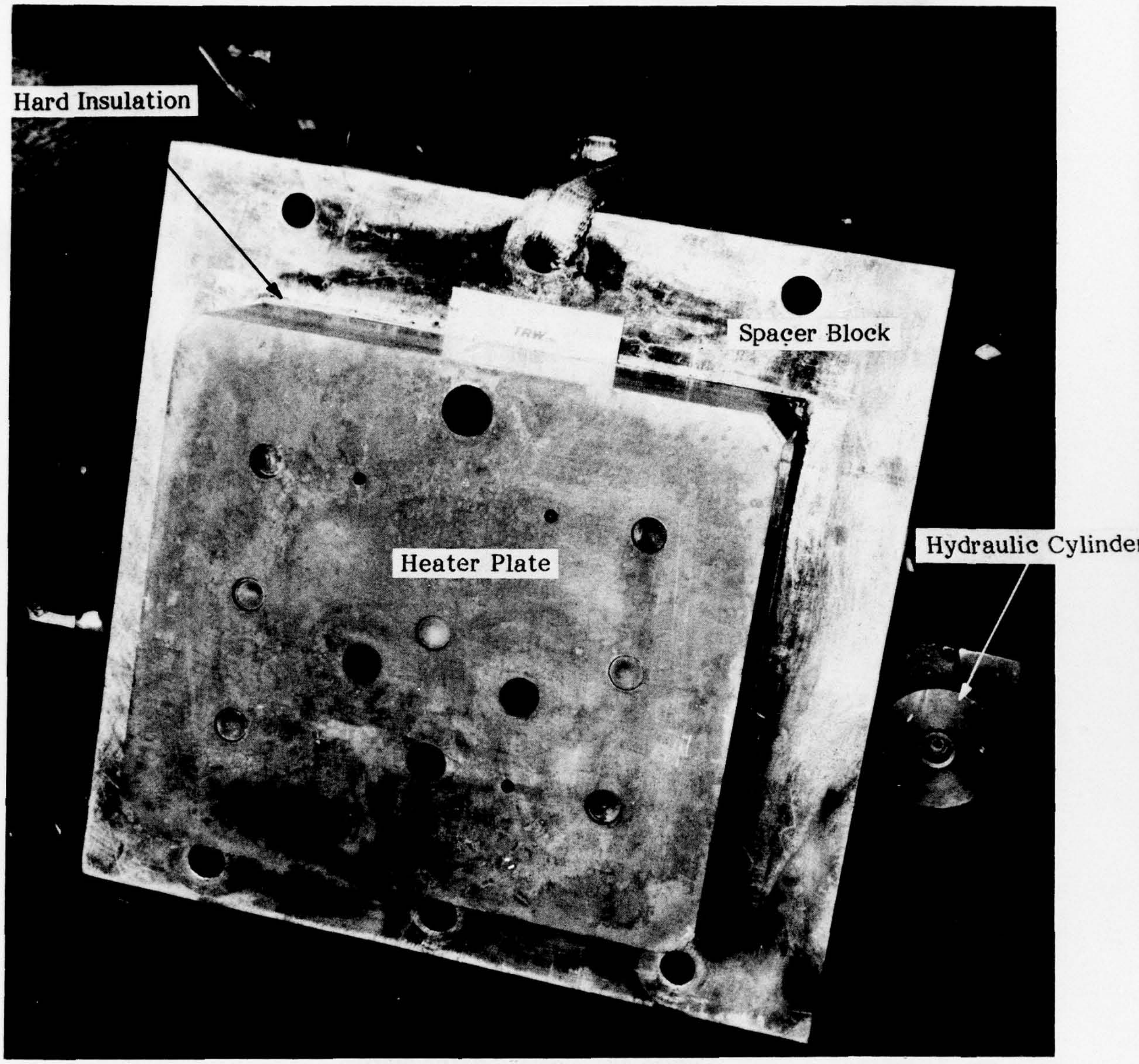


Figure 47. Upper tooling system showing some of the major components.

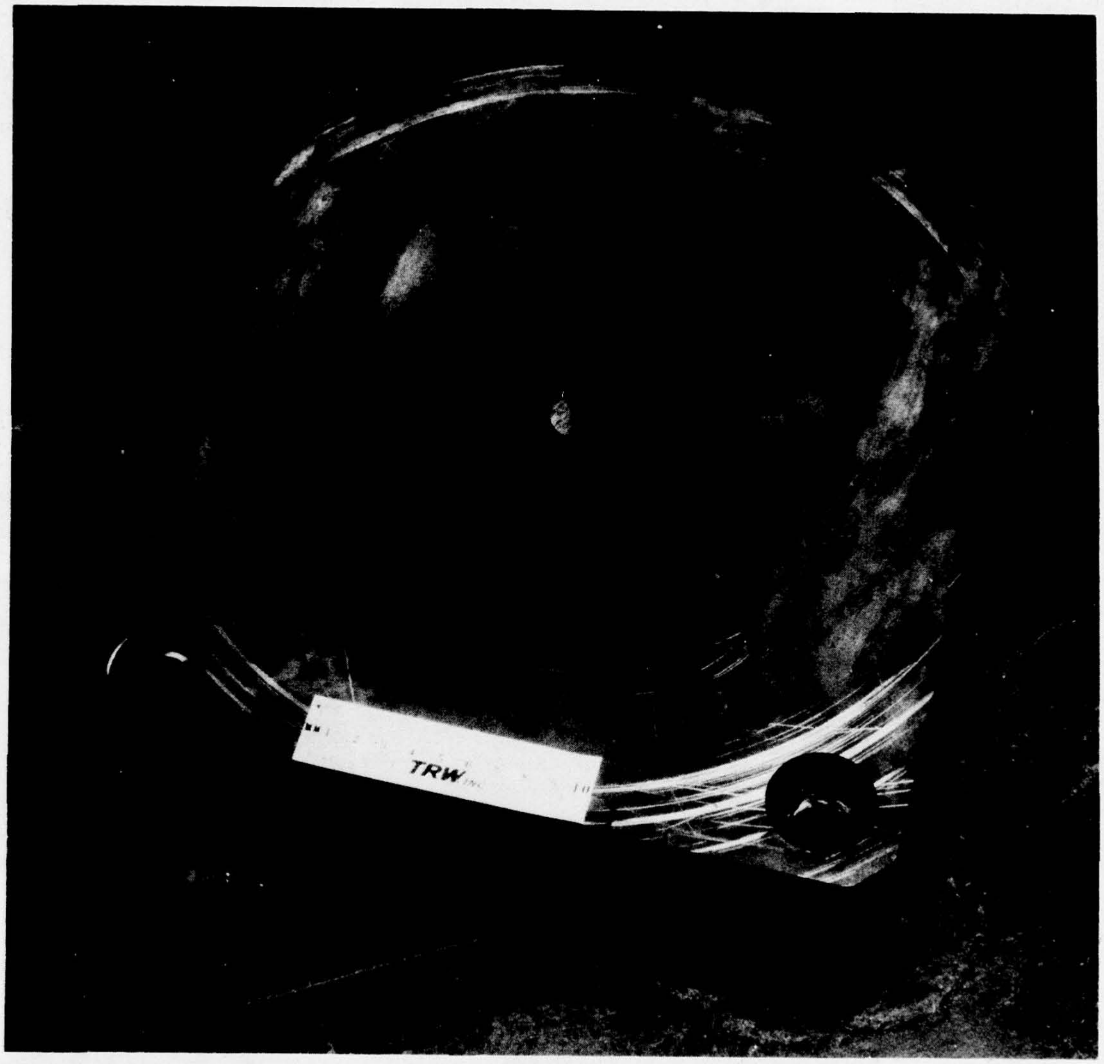


Figure 48. Lower heater plate.

to the heater plates were the modified TRW-NASA VIA cast die inserts which will be discussed in greater detail in a subsequent section. The IN-100 holder ring, shown in Figure 49, was used to provide positive alignment between the lower and upper die inserts and to also act as a heater ring. Eight heating rods 45.7-cm (18-inch) heated length, located horizontally, were used to heat the ring.

There were other components such as insulation, die ring support blocks, and clamps that also were a part of the tooling system and are illustrated in Figure 44. Of importance was the dimensional tolerance of all the holes used to locate the heating rods and wire feedthroughs, attachment bolts, ejector pins and mechanisms, and thermocouples. Four thermocouples were located in each heater plate and four thermocouples were attached to the surface of the die ring.

The tooling system was designed to operate at about 816^{o}C (1500^{o}F) with regards to thermal expansion and component shrink because of its proposed use on Contract F33615-76-C-5386; however, the tooling system was also operable in the temperature range 871-927^{o}C (1600-1700^{o}F), the temperature required to evaluate the trunnion die inserts.

The design of the form in the trunnion die inserts was based on the design of the part as described earlier. The designs of the inserts are shown in Figures 50 and 51. The inserts were mechanically attached to the remaining subassembly using U-700 bolts. A discussion of the casting of these inserts is presented in the following section. It should be noted that the ejector pin holes were machined into the upper die insert by electrical discharge machining (EDM), and were not cast into the insert block. IN-100 material was used for ejector pins and was machined in conjunction with the trunnion dies. A discussion of the casting and finish machining of the die inserts is presented in the following sections.

C. Die Insert Tooling Fabrication

1. Casting of Die Inserts

Vacuum induction melting and vacuum casting were used to produce the trunnion forge tooling die inserts. The die inserts were cast containing the major die cavity impressions. As will be discussed in the following section, final machining of the die configurations was conducted by EDM operations. The purpose of casting the die inserts with the major impression was to minimize finish machining costs and to obtain a more desirable or optimum casting microstructure at the surface and near-surface of the cavity than could be obtained in a die with the cavity machined entirely by EDM into a flat block. The optimized structure would result from faster cooling rates at the casting surface as opposed to the center of the cast blocks.

The ceramic casting molds were produced by Cast Masters, Inc., of Bowling Green, Ohio, using the Shaw process (63). They were then transported to the Cannon Muskegon Company, of Muskegon, Michigan, where they were vacuum cast from virgin master metal of the desired composition, also produced by Cannon Muskegon. The initial step in the production of the die inserts consisted of the preparation of split-half wooden models of the trunnion forging component. The wooden models were used to make the

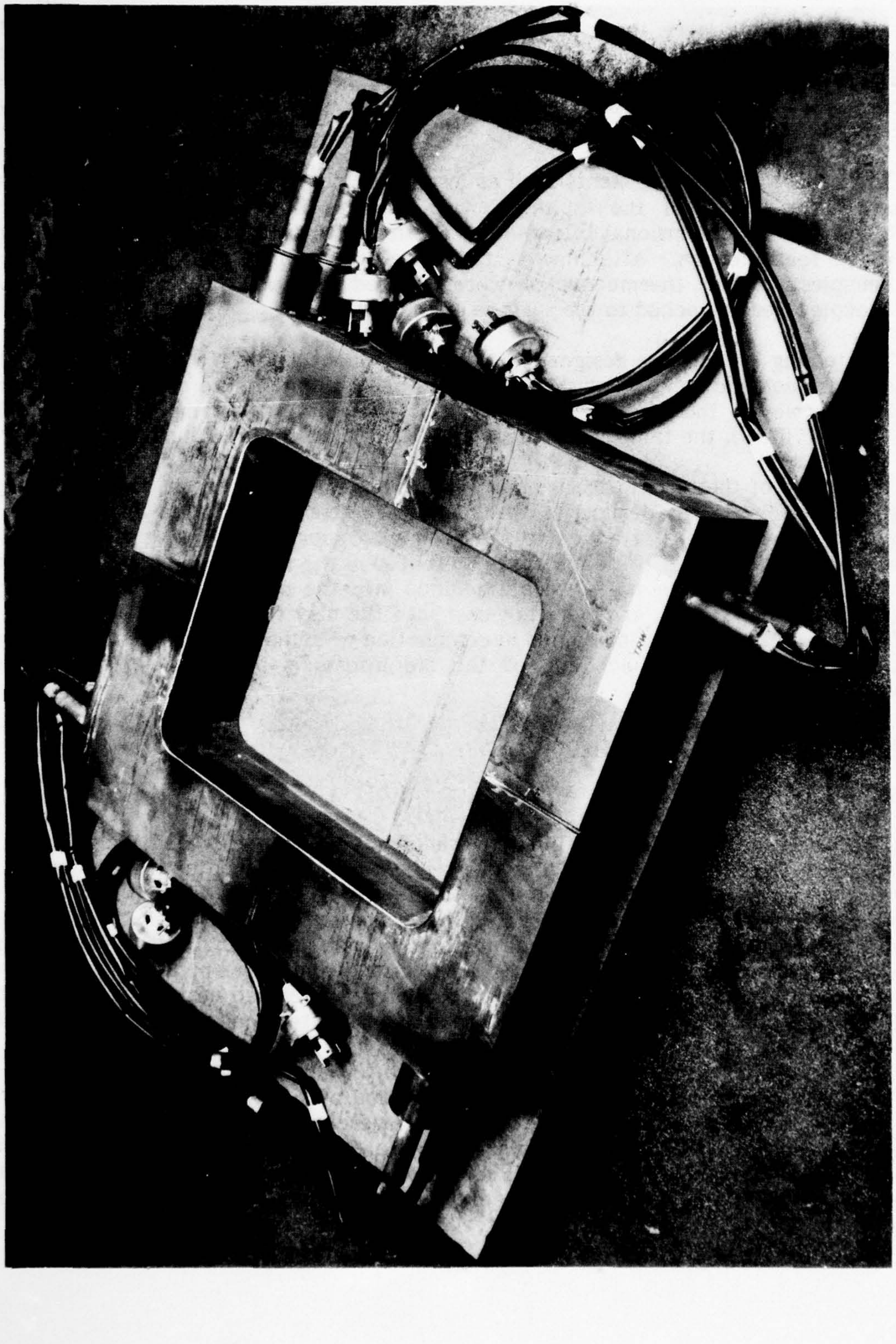


Figure 49. IN-100 die insert holder ring.

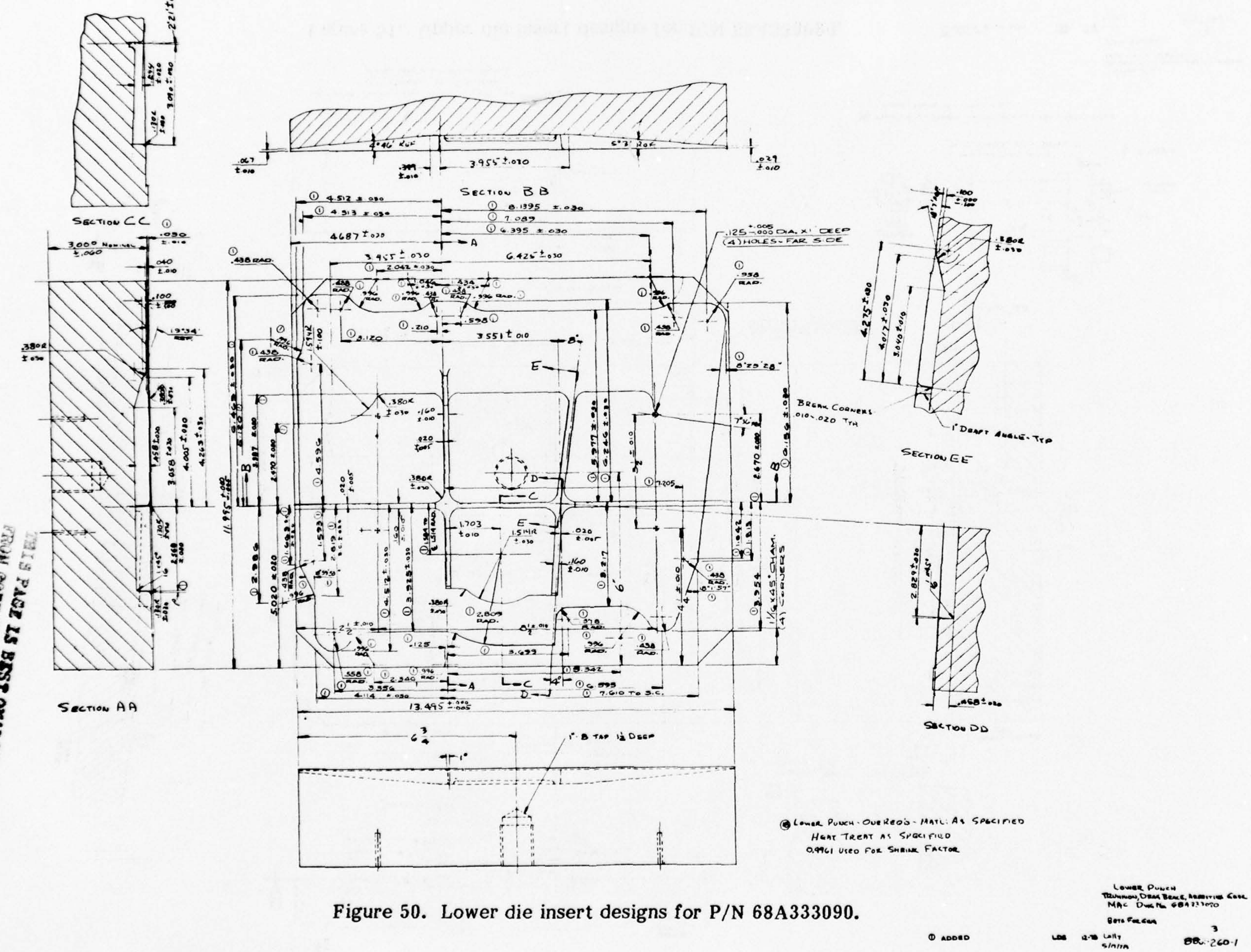


Figure 50. Lower die insert designs for P/N 68A333090.

79-226

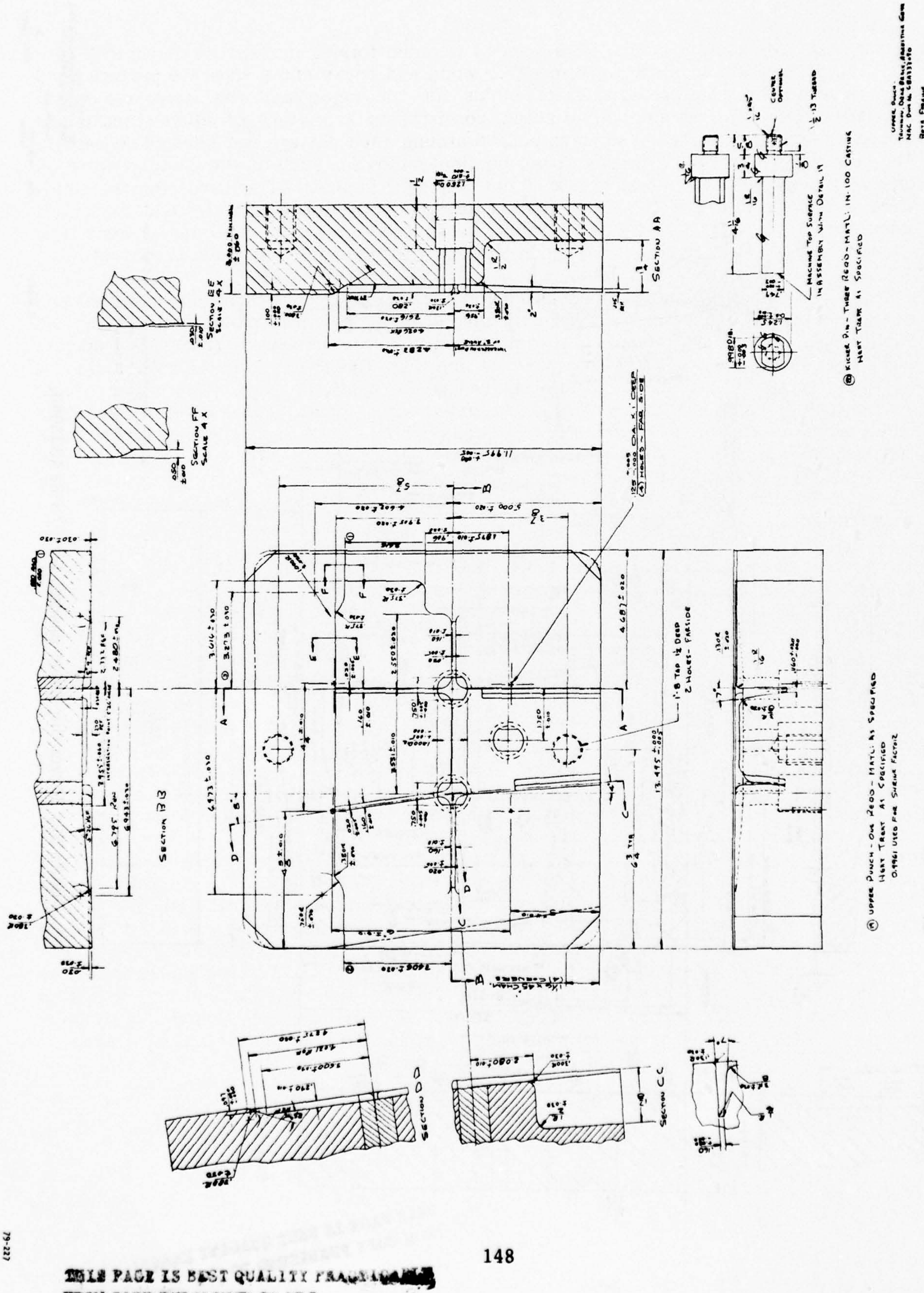


Figure 51. Upper die insert designs for P/N 68A333090.

79-227

patterns for the casting molds. The wooden trunnion forging models are shown in Figure 52. The patterns themselves were made of wood and epoxy and a separate pattern each was required for the preparation of molds for the upper and the lower die insert configurations. The ceramic mold slurry consisted of a mixture of ethyl silicate and zircon flour plus a hardener and was poured around each pattern and allowed to harden. The cope (upper portion) and drag (lower portion) molds for each of the castings were 66 cm (26 inches) x 61 cm (24 inches) x 10 cm (4 inches) in size. After hardening the molds were ignited to burn off the ethyl alcohol (present in the ethyl silicate) and the molds were fired at approximately $816^{o}C$ ($1500^{o}F$). The cope and drag mold halves were then transported by TRW personnel to Cannon Muskegon for assembly and vacuum casting.

Upon arrival at Cannon Muskegon the molds were oven dried, inspected and assembled by TRW personnel. Assembly involved clamping the molds and sealing the parting line with mold sealant. The castings were poured on successive days in Cannon's 135.7 kg (300 pound) vacuum induction melting furnace preheated overnight at approximately $260^{o}C$ ($500^{o}F$). Upon removal from preheat, the molds were inspected and the parting line and external cracks were sealed with mortar. Approximately 135.7 kg (300 pounds) of weights were placed on the cope portions of the molds to keep them down during pourings. Pouring was accomplished through a tundish with 8.9-cm (3-1/2 inch) diameter pour holes which discharged with the 5.1-cm (2-inch) diameter sprue. The central riser was covered with four layers of Fiberfax felt during pouring which acted as a melt shield and insulator.

Refining pressures were below one micron throughout both heats. The pour temperature for the upper die insert casting was $1443^{o}C$ ($2630^{o}F$), and for the lower die insert casting, $1449^{o}C$ ($2645^{o}F$). Pour time for each casting was approximately 2 minutes, and the pour weight for each was approximately 124.4 kg (275 pounds). Just prior to pouring the furnace was backfilled with 10 mm of argon. Immediately on pouring the furnace was vented, which took about 90 seconds, the mold removed and the riser hot topped with exothermic material. The castings cooled overnight in the molds before being shaken out. A photograph of the trunnion forge tooling castings immediately after mold shake-out is shown in Figure 53. Note the locations of the pour cup and riser. A photograph of the trunnion forge tooling showing detail of the as-cast upper die insert configuration is shown in Figure 54, while the detail of the lower die insert configuration is shown in Figure 55. As will be discussed in the following section concerning machining of the final die configurations, some surface irregularities (pitting and surface connected porosity) on the casting surfaces necessitated the removal of approximately 0.08 cm (0.03 inch) of surface material by EDM operations. There were no cracks observed on the casting surfaces. The Cannon Muskegon chemical analysis certification (in weight percent) of heat number VF-170, used to cast the trunnion forging dies, is listed in Table XXXVI. With the exception of chromium, all of the elements were within the modified TRW-NASA VIA specification range. The chromium content (5.7%) was slightly below the desired 5.8-6.2% range. The high purity of this material was reflected by the low oxygen and nitrogen contents, both at the 4 ppm level. The castings were shipped to TRW for removal of the gates and risers and were then ready for the final machining operations.

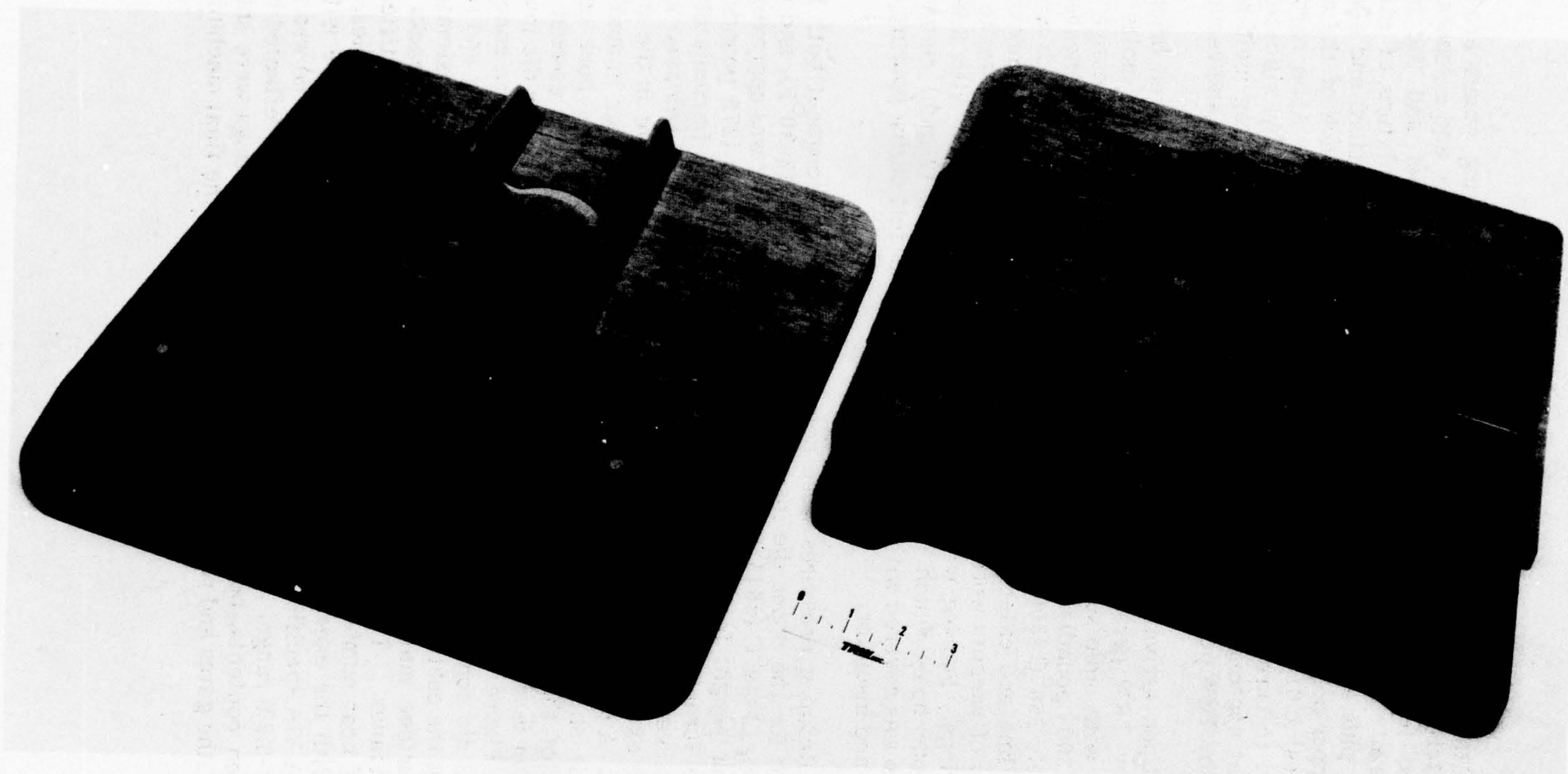


Figure 52. Wooden models of F-15 trunnion part used to make patterns for construction of the ceramic casting molds.

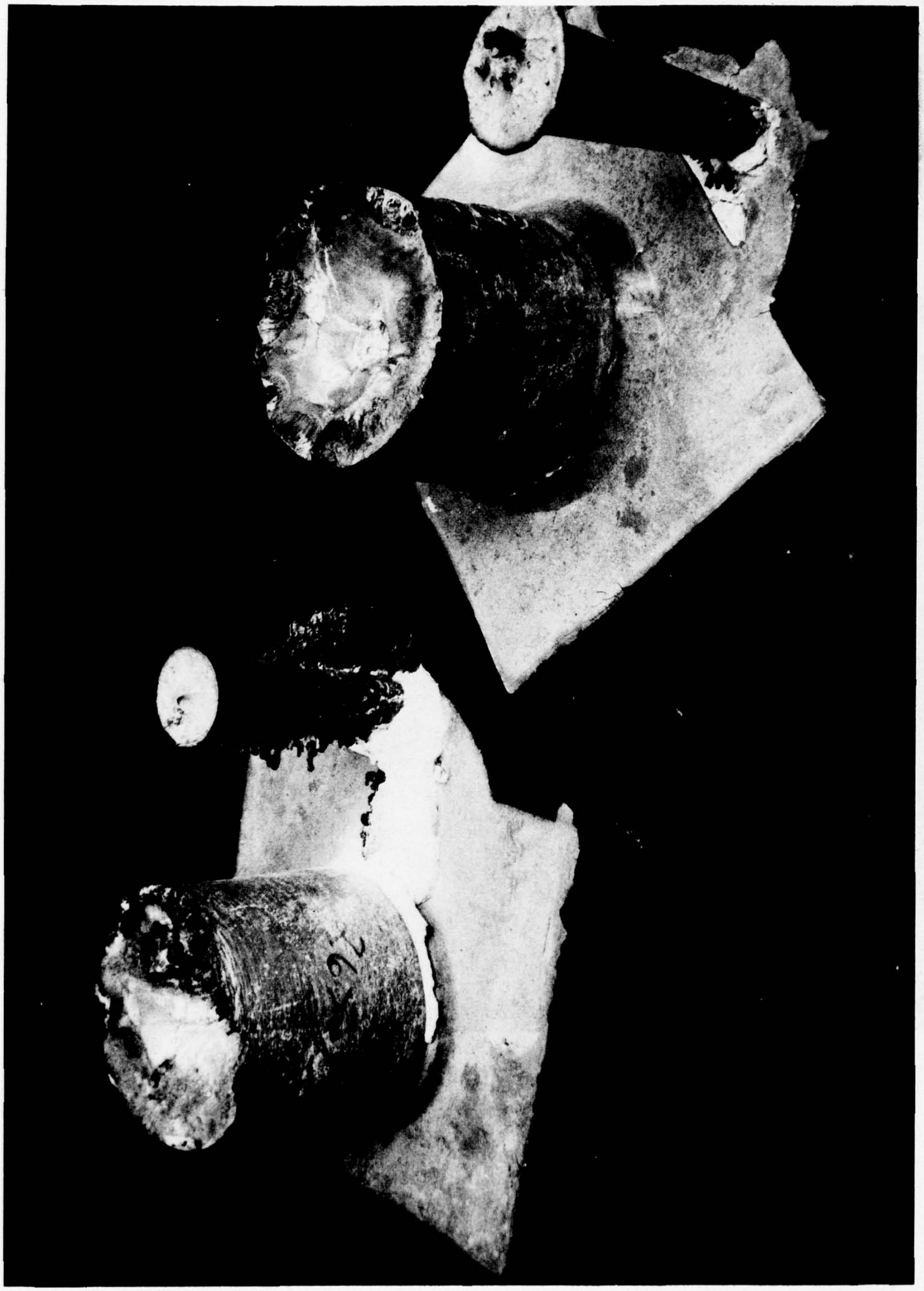


Figure 53. Photograph of trunnion forge tooling castings immediately after mold shake-out, showing location of pour cup and riser.

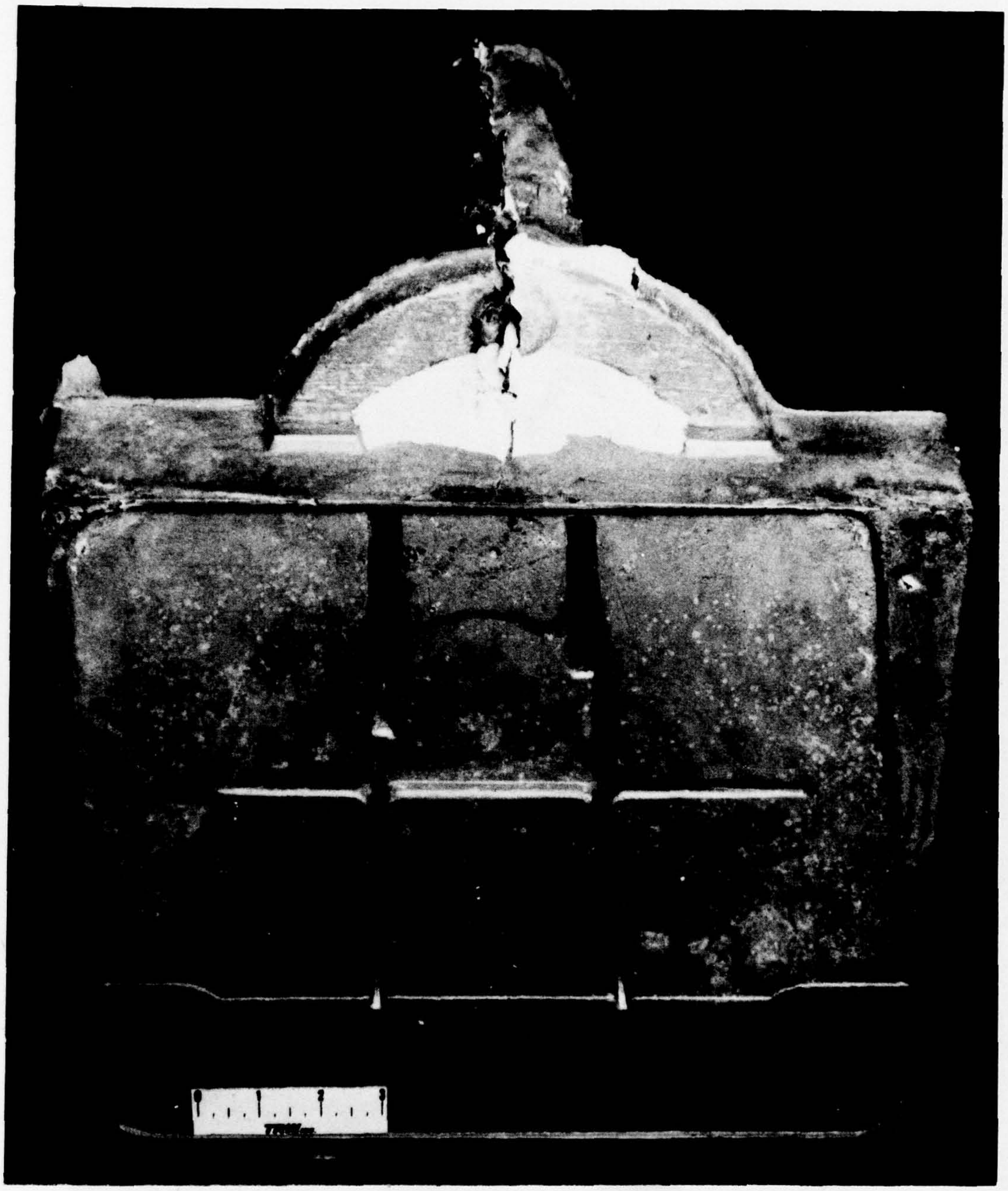


Figure 54. Photograph of trunnion forge tooling showing detail of the as-cast upper die insert configuration.

Figure 55. Photograph of trunnion forge tooling showing detail of the as-cast lower die insert configuration.

Table XXXVI

Chemical Analysis Certification (in Weight Percent) of Cannon Muskegon Heat Number VF-170, Used to Cast Trunnion Forging Dies[1]

Element	TRW-NASA VIA Modification V Specification	Heat VF-170 Analysis
C	.03-.05	.04
Al	5.4-5.8	5.40
B	.006-.010	.008
Cb	.4-.6	.4
Co	7.4-7.6	7.5
Cr	5.8-6.2	5.7
Hf	1.8-2.3	2.1
Mo	1.9-2.1	1.9
Ta	6.0-7.0	6.6
Ti	.9-1.0	.97
W	5.5-6.0	5.5
Zr	.01-.03	.03
Re	.20-.22	.20
O_2	25 ppm maximum	4 ppm
N_2	25 ppm maximum	4 ppm
Ni	Balance	

(1) Heat VF-170 was 100% virgin material.

2. Machining of Cast Die Inserts

The die inserts with the major die cavity impressions produced during casting are shown after finishing machining in Figure 56. Casting of the die with a cavity impression caused some unique problems and additional costs for the machining vendor compared to machining a cavity into a flat block. The exact position of the cavity had to be related to the sides of the die inserts prior to "squaring up" of the insert by finish grinding. Detailed inspection of the cavity was required prior to removing a specified amount of material from each side. Normally cast inserts with no cavity detail need only be finish machined, length and width direction, to the required dimensions of the blueprint with no prior detailed inspection. In addition, only the final insert dimensions were critical, while the amount of material removed from each side was not important as long as the blueprint, length and width, dimensions were met.

The finishing machining of the die impressions was conducted solely by EDM at contoured Electrode, Mentor, Ohio. After the sides of the blocks were finish ground a reinspection of the die impressions indicated an approximate .25 cm (0.1 inch) mismatch between the inserts. It is not known whether the mismatch of the die impressions occurrred due to an error in inspection by the EDM vendor or an error in machining by the grinding vendor. There was also some question afterwards as to the accuracy of the location of the impressions after casting to allow sufficient material for finish machining of the sides such that the sides were dimensionally correct with respect to the cavities. It should be noted that although casting dies with impressions is not unique, it is new to the isothermal forging of structurals business. In addition, the experience by the casting vendors and machining vendors is limited to date for the types of alloy being cast.

Although the problem arose with respect to the mismatch, the die impressions were found to be produced quite accurately. The upper die insert could have been left as cast in all flat surface detail; however, because of surface irregularities (porosity and pitting) a nominal 0.08 cm (0.03 inch) was removed by EDM. The ribs had about 0.08 cm (0.03 inch) removed from each side, but were sunk an additional 0.2 cm (0.08 inch) in depth for the shallow ribs and 3.2 cm (0.125 inch) for the two deep ribs. During pattern manufacture there was some concern as to the depth of the rib to allow for without problems in mold breakage occurring at a later date. The decision was to specify shallow ribs. Machining of the bottom die insert could have been conducted similarly except as discussed previously there was a mismatch between the detail in the two cavities. There was also a difference of 0.06 cm (0.025 inch) between the measurement of the distance between ribs and the blueprint requirement. This difference probably would have been absorbed during an EDM "clean up" operation similar to the one performed on the upper die insert. The entire cavity was resunk approximately 1.0 cm (0.4 inch). The lower die insert was selected to be entirely resunk because of its shallow ribs compared to the two deep ribs in the upper die insert.

Machining of die inserts with cast-in impressions can be cost effective. The cost savings realized is a direct result of the reduced number of EDM electrodes required and the labor in finish machining. Some of this savings was offset in the present effort by the additional inspection required by the machining vendor. Another consideration is the cost in producing patterns and molds. An accurate cost analysis of this approach would require that the vendors become more experienced in their areas in dealing with cast-in impression dies or die inserts.

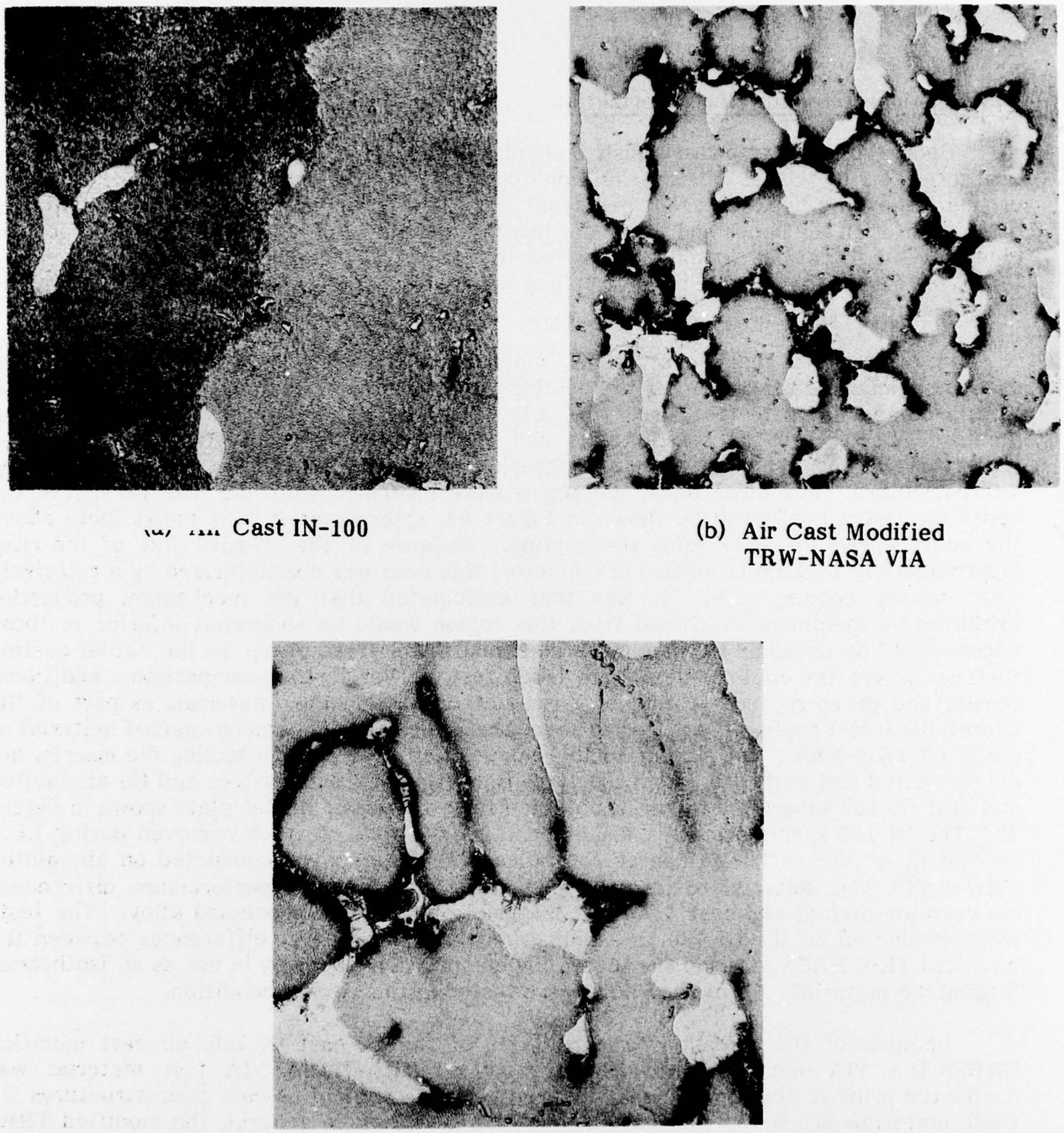
Figure 56. Photograph of completely machined trunnion forge tooling die inserts. Note the ejection pins in the upper die insert (left hand casting). The lower die insert is shown in the right hand casting.

D. Die Insert Tooling Characterization

Upon the completion of finish machining operations, the forging die inserts were characterized by visual observation, photography, dimensional inspection of selected critical areas and by fluorescent penetrant inspection. Dimensional analysis of a number of areas on both the upper and lower die inserts indicated that the measurements met the blueprint specifications within the desired tolerances. Fluorescent penetrant inspection failed to reveal the presence of any surface connected porosity, cracking or other surface irregularities.

In addition to these evaluations, mechanical property testing was conducted on specimens machined from one of the castings to characterize this material in comparison to the compositions tested during the alloy development efforts of Phase I. Testing included 927°C (1700°F) tensile tests and 954°C (1750°F)/206.8 MPa (30 ksi) creep-rupture tests as well as a metallographic analysis to characterize the casting microstructure. For these tests, specimens were machined from the riser portion of the upper die insert configuration shown in Figure 54, approximately 2.54 cm (1 inch) above the point where the riser joins the casting. Because of the massive size of the riser (approximately 17.8 cm (7 inches) in diameter) this area was characterized by a relatively slow casting cooling rate. It was thus anticipated that the mechanical properties exhibited by specimens machined from this region would be somewhat inferior to those which would be obtained by specimens machined from areas closer to the actual casting surfaces where the cooling rates were much faster. For further comparisons, additional tensile and creep-rupture testing was conducted on two other materials as part of the overall die insert tooling characterization. These included (1) vacuum-melted material of modified TRW-NASA VIA similar to the composition of the forge tooling die inserts, but air re-melted and cast into a keel block configuration at Cast Masters and (2) air-melted and cast IN-100 superalloy material obtained from the lower heater plate shown in Figure 48. The IN-100 specimens were machined from the rods of stock removed during EDM machining of the cartridge heater rod holes. The tests were conducted on air-melted TRW-NASA VIA material to identify any mechanical property performance differences between air-melted and cast VIA type material and vacuum processed alloy. The tests were conducted on the IN-100 to establish mechanical property differences between the modified TRW-NASA VIA and the superalloy composition currently in use as an isothermal forging die material. All of the alloys were tested in the as-cast condition.

Analysis of the as-cast microstructures of the air-cast IN-100, air-cast modified TRW-NASA VIA and the vacuum-cast modified TRW-NASA VIA test material was conducted prior to the mechanical property testing. Typical as-cast microstructures for these materials are shown in Figure 57 and indicate that, in general, the modified TRW-NASA VIA structures are characterized by larger and more numerous eutectic type gamma-prime colonies than those present in the IN-100 material. The colonies within the VIA structures also appear more interconnected that the isolated colonies in the IN-100 microstructure. Comparison of Figure 57a with the microstructure for the Phase I IN-100 material (vacuum cast) shown in Figure 19c indicates the presence of considerably fewer gamma-prime eutectic colonies as well as intragranular carbide precipitates in the air-cast material. These differences were primarily the result of section size effects caused by variations in the casting cooling rates in the material. The air-cast IN-100 specimen was prepared from the massive heater plate casting, Figure 48, while the Phase I vacuum-



(a) Air Cast IN-100

(b) Air Cast Modified TRW-NASA VIA

(c) Vacuum Cast Modified TRW-NASA VIA

Figure 57. Light photomicrographs of typical as-cast microstructure of (a) air-cast IN-100 heater block, (b) air-cast modified TRW-NASA VIA keel block casting and (c) vacuum-cast modified TRW-NASA VIA trunnion forging die casting. Magnification, 100X.

cast specimen was prepared from the corner of a cast block. Comparison of Figures 57b and c with Figure 25 showing the microstructures of the TRW-NASA VIA Modification IV and V alloys (the compositions from which the forge tooling die insert casting alloy was formulated) indicates an overall similarity in the types of microconstituents present throughout the structures. All four materials were characterized by rather large gamma-prime eutectic colonies located primarily in interdendritic regions and, in general, these colonies were usually interconnected with a dark etching microconstituent. The major differences in the microstructures of the air-cast and the various vacuum-cast materials were primarily a reflection of the differences in casting cooling rates for the different geometries. The air-cast keel block, at approximately 13.8 kg (30 pounds) and the rectangular vacuum-cast blocks, at approximately 22.6 kg (50 pounds) were much smaller in size than the 124.4 kg (275 pounds) die insert casting. As a result, the keel block specimen, Figure 57b and the specimens from the cast blocks, Figure 25, exhibited microstructures characterized by a finer detail than the specimen prepared from the riser portion of the forging die insert, Figure 57c. In particular, colonies of primary gamma-prime eutectic were somewhat smaller in size than those observed in the riser material and also appeared much closer to one another. This was characteristic of the faster cooling rates in the keel block than in the riser portion of the forging die inserts. Higher magnification analysis of the gamma-prime particles precipitated upon solidification also indicated that the keel block and casting block specimen were characterized by smaller particles in a finer dispersion than was present in the vacuum-cast riser material.

The results of the 927°C (1700°F) tensile tests are listed in Table XXXVII and indicate that the air-cast modified TRW-NASA VIA material exhibited the highest ultimate tensile and 0.2% offset yield strengths of the three materials tested. At an average yield strength of 639.9 MPa (92.8 ksi), the air-cast material was considerably better than the vacuum-cast modified VIA, which exhibited an average yield strength of 525.4 MPa (76.2 ksi). The IN-100 was quite poor in comparison, with an average yield strength of only 379.9 MPa (55-1 ksi). This was also poor in comparison to the IN-100 (vacuum-cast) tested in the Phase I alloy development effort, which exhibited an average yield strength of 437.2 MPa (63.4 ksi), Table XIII. An analysis of the Phase II tensile data in comparison to the yield strength results presented in Figure 40 for the best of the powder metallurgy, casting superalloy and metastable carbide alloys indicates that the air-cast TRW-NASA VIA modification also exhibited the highest yield strength values obtained throughout the entire alloy development effort. The 574.4 MPa (83.3 ksi) average yield strength value exhibited by the powder metallurgy Modification II alloy and the 541.3 MPa (78.5 ksi) average yield strength exhibited by the casting superalloy Modification V alloy were both inferior to the keel block material. It was significant to note, however, that specimens machined from the riser portion of the vacuum-cast die insert tooling did exceed the program goal of 517 MPa (75 ksi) and were quite close to the best of the casting alloys studied in Phase I 525.4 MPa (76.2 ksi) versus 541.3 MPa (78.5 ksi) in spite of the slow cooling rates. Because of the faster cooling rates at the surfaces of the forging die castings, much higher strength properties would be expected of specimens machined from these locations. It was clear, moreover, that in terms of yield strength the vacuum-cast modified TRW-NASA VIA alloy was superior to the currently used VIA alloy was superior to the currently used IN-100 isothermal forging die alloy composition.

Table XXXVII

927°C (1700°F) Tensile Test Results for Cast Materials Used to Characterize Trunnion Forge Tooling[1]

Alloy	Ultimate Tensile Strength MPa	Ultimate Tensile Strength Ksi	0.2% Offset Yield Strength MPa	0.2% Offset Yield Strength Ksi	% Elongation	% Reduction Area
Air Cast[2] IN-100	499.9	72.5	388.9	56.4	3.9	2.2
	411.0	59.6	370.3	53.7	5.0	3.1
Air Cast[3] TRW-NASA VIA Modification V	722.0	104.7	674.4	97.8	2.5	1.2
	688.9	99.9	608.8	88.3	5.3	9.3
Vacuum Cast[4] TRW-NASA VIA Modification V	639.2	92.7	521.3	75.6	2.0	2.0
	625.4	90.7	528.9	76.7	3.6	3.9

(1) As-cast properties from 0.6 cm (0.252 inch) diameter test specimens.

(2) Machined from heater block casting.

(3) Machined from 13.6 kg (30 pound) keel block casting.

(4) Machined from riser portion of cast trunnion forge tooling die block.

The results of the 954°C (1750°F)/206.8 MPa (30 ksi) creep-rupture tests are listed in Table XXXVIII. Unlike the tensile results, the vacuum-cast modified TRW-NASA VIA was superior to the air-cast material and the IN-100. Rupture lives averaged 148.6 hours for the vacuum-cast material, compared to the 99.9 and 44.9 hour average exhibited by the air-cast modified TRW-NASA VIA and the IN-100, respectively. Time to reach 0.1% creep was also superior for the vacuum-cast material, ranging from 2.27-15.6 hours, compared to the 1.7-4.5 hour range for the air-cast alloy. The IN-100 specimens all reached 0.1% creep in less than 0.63 hours. In addition to being poor compared to the other Phase II materials, the air-cast IN-100 was also poor in comparison to the vacuum-cast material evaluated in Phase I. As shown in Table XIV, the Phase I IN-100 averaged 104.4 hours rupture life and exhibited time to reach 0.1% creep ranging from 1.99 to 4.38 hours. An analysis of the Phase II creep-rupture data in comparison to the results obtained for the casting alloys tested in the Phase I alloy development efforts indicated that the specimens machined from the riser portion of the vacuum-cast modified TRW-NASA VIA exhibited generally equivalent creep-rupture properties. In terms of rupture life, for example, the maximum value was exhibited by a specimen from the Series I TRW-NASA VIA composition at 187.6 hours (Table XIV), while the vacuum-cast material exhibited a maximum of 197.2 hours. In terms of time to reach 0.1% creep, the maximum value was exhibited by the Modification V alloy at 18.2 hours (Table XX) while the vacuum-cast material exhibited a maximum of 15.6 hours. A particularly important aspect of these property levels was that they were achieved by material obtained from the riser portion of the forging die tooling. Because of the faster casting cooling rates at the forging die surfaces, it is anticipated that the creep-rupture properties there would be superior to those achieved by the riser material. Of potentially greater significance, however, was the creep-rupture property improvements of the vacuum-cast modified TRW-NASA VIA material in comparison to the currently used IN-100 isothermal forging die material. The average rupture life of the TRW-NASA VIA material was three times that of the IN-100 and the time to reach 0.1% creep was an order of magnitude greater than that of the IN-100.

Table XXXVIII

$954^{\circ}C$ $(1750^{\circ}F)$/206.8 MPa (30 Ksi) Creep Rupture Test Results for Cast Material Used to Characterize Trunnion Forge Tooling(1)

Alloy	Hours to Failure	Hours to 0.1% Creep	% Elongation	% Reduction Area
Air Cast IN-100(2)	37.8	0.20	4.1	2.7
	37.3	0.47	5.0	4.3
	59.7	0.63	5.0	4.7
Air Cast TRW-NASA VIA Modification(3)	110.8	4.5	2.8	3.5
	89.0	1.7	2.2	2.3
Vacuum Cast TRW-NASA VIA Modification(4)	140.8	4.33	5.1	7.0
	197.2	15.60	5.7	3.5
	107.8	2.27	3.7	3.5

(1) As-cast properties from 0.6 cm (0.252 inch) diameter test specimens.

(2) Machined from heat ring casting.

(3) Machined from 13.6 kg (30 pound) keel block casting.

(4) Machined from riser portion of cast trunnion forge tooling die block.

V SUMMARY AND CONCLUSIONS

A program was conducted in order to develop materials and their required processing techniques for the production of isothermal forging dies exhibiting higher yield strength and increased resistance to dimensional distortion at a reduced overall cost than currently available with present-day nickel-base superalloy and molybdenum alloy compositions. Target property goals for the new die materials included 517 MPa (75 ksi) 0.2% yield strength at 927^{o}C (1700^{o}F) and a time in excess of 30 hours to reach 0.1% plastic creep at 954^{o}C (1750^{o}F)/206.8 MPa (30 ksi). To accomplish this, efforts were directed towards evaluating the applicability of powder metallurgy techniques and improved casting techniques for the production of die cavity shapes from new superalloy compositions. The technical approach involved two phases. In Phase I - Alloy Development, efforts included the evaluation of nine powder metallurgy, nine casting alloy and nine metastable carbide alloy compositions in three series of alloys each. Evaluation included mechanical property screening tests on the first two series of alloys and a more complete property evaluation on the two most promising compositions in the final series. The more complete evaluations included tensile, creep-rupture and thermal fatigue tests as well as lubricant compatibility tests with typical isothermal forging lubricant compositions. IN Phase II - Die Fabrication, the most promising die material/process combination was used to fabricate tooling for the F-15 trunnion drag brace arresting gear. The performance of this tooling will be evaluated at TRW during the subsequent production of a series of isothermally forged parts under Air Force Contract F33615-76-C-5386, "Isothermal Forging Beta Titanium."

For the Phase I - Alloy Development study, while the first series of alloys for each of the processing methods included TRW-NASA VIA, TAZ-8A, IN-792 and IN-100, subsequent efforts were directed towards modifications of the TRW-NASA VIA chemistry. The optimum alloys for each processing method were thus based on the TRW-NASA VIA composition. The powder metallurgy (Modification II) alloy featured a higher hafnium level compared to the base composition (2.29% versus 0.51%), tantalum reduced from 9.0% to 5.9% and had rhenium added at the 0.23% level. Two casting superalloys (Modification IV and V) exhibited comparable properties and were quite similar in composition to the powder metallurgy alloy. The best metastable carbide alloy was the Series I composition, differing from the original TRW-NASA VIA composition in that rhenium was removed from the alloy and carbon was added in the form of metastable SiC particles during attrition of the base alloy powders.

Die alloy/process selection was made primarily on the basis of the tensile and creep rupture properties exhibited by these alloys as well as by considerations of the lubricant compatibility and thermal fatigue results. Analysis of the tensile strength results indicated that the values were comparable for the three processing methods. In terms of average values, all of the alloys met the program yield strength goal suggesting that all three alloy/processing combinations exhibited sufficient potential for application as isothermal forging die materials. In contrast, the creep-rupture results indicated a considerable difference between the casting alloys and the powder metallurgy and metastable carbide alloy compositions. This was manifested primarily in terms of time to

reach 0.1% creep and in rupture life. For the casting superalloys, for example, the times to reach 0.1% creep were approximately double those of the powder metallurgy materials (18.2 hours maximum versus 9.5 hours maximum) and the average rupture lives were approximately six times those of the powder metallurgy materials (120 hours versus 19.8 hours). While the times to reach 0.1% creep for the casting alloys were short of the 30 hours program goal, they did offer considerable improvement over the properties available in IN-100 (the currently used nickel-base superalloy forging die material) cast in similar thick sections. These results suggested that only the casting superalloys exhibited sufficient potential for application as isothermal forging die materials.

The die alloy/process selection included the lubricant compatibility and thermal fatigue results for only the casting alloys because these materials exhibited the best overall potential for isothermal forging die applications. The lubricant compatibility test results with the OPT 112 and the Deltaglaze 69 lubricants indicated that both of the Modification IV and V alloys exhibited comparable resistance to attack and further that they were superior in resistance compared to the currently used IN-100 forging die material. The thermal fatigue results indicated that the Modification IV alloy, containing the higher hafnium content (2.1% versus 1.78%) and the higher tantalum content (8.79% versus 6.79%) was the best of the two casting alloys. On the basis of the fact that both alloys exhibited comparable tensile, creep rupture and lubricant compatibility results, and that Modification IV was superior to Modification V in terms of thermal fatigue, the chemistry range selected for the die fabrication efforts included the Modification IV alloy, with tantalum lowered from approximately 9.0% to a range of 6.0-7.0% because of the current high costs for tantalum. The chemistry range included the following in weight percent:

C	0.03 - 0.05
Al	5.40 - 5.80
Ti	0.90 - 1.00
Co	7.40 - 7.60
Cr	5.80 - 6.20
Hf	1.80 - 2.10
Mo	1.90 - 2.10
Cb	0.40 - 0.60
Ta	6.00 - 7.00
W	5.50 - 6.00
B	0.006 - 0.010
Re	0.20 - 0.22
Zr	0.01 - 0.03
O_2	25 ppm maximum
N_2	25 ppm maximum

For the Phase II - Die Fabrication efforts, the hot die forging system was built using a modular tooling design concept which allows the use of a support system for several different production parts, thus requiring only the replacement of the die and punch assembly for a particular configuration. The die and punch are thus considered as die inserts and were made from the modified TRW-NASA VIA casting composition. Vacuum induction melting and vacuum casting were used to produce the tooling with the major die cavity impressions cast directly into the inserts. EDM machining was used to produce the finished die impressions. While a mismatch was discovered between the die inserts, prior to finish machining, the die impressions themselves were found to be produced quite accurately. Because of surface irregularities (porosity and pitting), however, a nominal 0.08 cm (0.03 inch) was removed by EDM. Die insert tooling characterization included tensile and creep rupture tests on specimens from the riser portion of one of the castings. For comparative purposes, tests were also conducted on air-cast TRW-NASA VIA of similar composition and on air-cast IN-100.

For this effort the IN-100 specimens were machined from material comparable to the vacuum-cast TRW-NASA VIA in section size thickness. The air-cast TRW-NASA VIA, on the other hand, was machined from relatively thin section size material. The 927°C (1700°F) tensile results indicated that the vacuum-cast modified TRW-NASA VIA was inferior to the air-cast material, but still met the program yield strength goal and was, in general, comparable to the compositions evaluated during the Phase I effort. The air-cast alloy exhibited an average yield strength of 574.4 MPa (833.3 ksi) compared to 525.4 MPa (76.2 ksi) for the vacuum-cast alloy. The 954°C (1750°F)/206.8 MPa (30 ksi) creep results indicated that the vacuum-cast material was superior to the air-cast alloy in spite of the section size differences and was also comparable to the compositions evaluated during the Phase I study. Of potentially greater significance, however, were the mechanical property improvements of the vacuum-cast modified TRW-NASA VIA material in comparison with the currently used IN-100 isothermal forging die material. The average yield strength of the TRW-NASA VIA was 524.5 MPa (76.2 ksi) compared to the 379.9 MPa (55.1 ksi) average for the IN-100 material. The average rupture life of the TRW-NASA VIA was three times that of the IN-100 and the time to reach 0.1% creep was an order of magnitude greater than that of the IN-100. These mechanical property results, in conjunction with the improved lubricant compatibility of the modified TRW-NASA VIA compared to IN-100, suggested strong potential for the applicability of this alloy as an isothermal forging die material.

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